COLD FUSION

Official thumbs down

Washington

To no one's surprise, the panel set up by the Department of Energy (DoE) to assess cold fusion has sent in a negative report. An interim opinion released earlier this year by the panel, a group of 23 electrochemists, nuclear physicists and materials scientists assembled by the DoE's Energy Research Advisory Board (ERAB), acknowledged that there were some odd and unexplained results in the air but recommended against any special support of cold fusion experiments (see *Nature* **340**, 174; 1989). The panel's final report expresses the same verdict a little more forcefully, but the ERAB meeting at which the report was approved and passed on to DoE Secretary James Watkins did reveal some differences of opinion on the significance and credibility of the handful of 'positive' results still extant.

Summarizing the final report, John Huizenga, a professor of physics and chemistry at the University of Rochester and one of the ERAB panel's co-chairmen, showed little patience with those who have continued to claim that there is at least a little evidence for cold fusion of the heat-producing variety. None of the calorimetry, he declared, was of good enough quality to be believed, and in any case calorimetry alone would never prove the case. Huizenga was particularly unhappy with those who argued that because the claimed excess heat could not possibly be of conventional origin it had to be nuclear, despite measurements which showed unequivocally that no fusion products were being generated.

Discounting the calorimetry, the only anomalous claim still needing an explanation was that of Kevin Wolf of Texas A&M University, who repeatedly finds tritium in his electrochemical cells. But new evidence from Wolf, discussed at last week's ERAB meeting, argues against a nuclear origin. Tritium produced by deuterium fusion comes with an energy of 1 MeV and is accompanied by a 3 MeV proton. Energetic tritons will react with deuterium, either in the palladium electrode or in the electrolyte, producing secondary neutrons, and the protons can excite some palladium nuclei, which give up a 500 keV photon on de-excitation. In his latest experiments, Wolf has searched for both these secondary signatures and found nothing. His tritium, therefore, must have some kind of low-energy origin.

Nevertheless, Wolf has also searched minutely for sources of contamination, and found nothing there either.

This puzzling result, together with a few calorimetric measurements that he found somewhat convincing, led recent Nobel prizewinner Norman Ramsey of Harvard University (see *Nature* **341**, 556; 1989), the panel's other co-chairman, to conclude that the case against cold fusion was not closed. At his insistence, a preamble was added to the report's conclusions which notes that "it is not possible to state categorically that all the claims for cold fusion have been convincingly either proved or disproved".

Huizenga felt that this preamble "weakened" the report, but Ramsey and some others felt that the report would undermine itself if it looked too dogmatic in its dismissal of the evidence.

On the subject of the low-level fusion process espoused by Steven Jones of Brigham Young University, the panel agreed that more work was needed, although here again Huizenga, noting that all the claims were of marginal statistical significance, seemed less impressed than some of the other panel members that there really was a phenomenon to be explained.

Although he showed some sympathy for those still making claims for cold fusion, Ramsey also said that if the results on which the claims were based came from his laboratory, he would keep quiet and do more experiments until he understood better what was going on. The ERAB report accepts that people are free to submit experimental proposals to granting agencies in the hope of obtaining support for cold fusion experiments, but recommends that only if experiments are likely to yield useful results — closed-cell calorimetry with simultaneous nuclear monitoring is deemed essential — should support be given. **David Lindley**

EAST GERMAN EXODUS

Problems remain for those who stay

Munich

East German researchers now living in West Germany are happy but sceptical about the events in East Germany that culminated in last week's opening of the Berlin Wall. No one disputes that the opening of the borders is a momentous and irreversible step, but many doubt that the effects of 40 years of Communist repression can be wiped away overnight: the repressive system is more deeply entrenched in East Germany than, for example, in Hungary, which has also enacted radical reforms this year.

Under the old system, said a number of researchers, Party members and other desirables were given the opportunity to travel abroad, publish in foreign journals and make progress in their scientific careers, but those who lacked an ideological pedigree were cut off from the West and forced to engage in political activities against their will. The pessimism brought on by such obstructions caused these people, like so many others, to escape.

Those who tried to leave by legal means found that as soon as they submitted visa applications, they were forbidden to work in their laboratories. One said he was reinstated only after an invitation to a scientific meeting from a prominent West German research institution, in which he now has a job. But the only way to find out about such opportunities is to have personal and professional contacts, said another.

Researchers said after the recent lifting of travel restrictions that they were waiting to see if the changes are large enough to encourage development of a system based on personal achievement and not privilege. Even so, East German research laboratories will still lack the money to order basic supplies, let alone to subscribe to foreign journals. Steven Dickman

West Germany continued to welcome all those who came over from East Germany on Friday, even though the capacity of the refugee camps had nearly been exhausted. Most of the refugees who had arrived before the Wall came down seemed to be in practical, not academic, professions, although an accurate tally was made impossible by the large numbers. But organizations involved in helping East German researchers and academics get on their feet in West Germany said they would need much more money to continue the process.

Uta Paffhausen of the Education Ministry reported that the number of applications for "adjustment aid", given to help East German emigrants find academic posts, had risen to about 50 in the past two months. The grants are provided by the ministry as well as by the Otto Benecke Foundation. The ministry also administers a programme to provide fellowships for refugees whose training is unsuitable for working in West Germany. The two programmes were to provide a total of DM 24 million ($13 million) in 1990 when the refugee total was still expected to be about 60,000. But by 10 November, the number had topped 500,000, including 200,000 East Germans and another 300,000 ethnic Germans from Poland and the Soviet Union. Photo: The wall near Checkpoint Charlie last Friday, seen from the East (AP).

Mechanism of solid-state fusion

SIR—Experiments by Derjaguin *et al.*[1,2] indicating that MeV neutrons were emitted during the destruction of heavy ice or monocrystals of LiD went almost unnoticed, whereas recent claims of 'cold fusion'[3,4] have attracted worldwide attention, ranging from highly enthusiastic confirmations to equally sceptical rejections. Here we wish to present some relevant considerations of mechanisms which could enhance nuclear interactions in the solid state.

A body of experimental evidence[5,6] suggests that hydrogen resides in transition metals as a simple 'lattice gas'. Although the rate for nuclear fusion of d + d in the lattice may be increased over the 'free' rate because of screening of the repulsive Coulomb potential by lattice electrons, the enhancement cannot be large enough[7] to explain the claims of refs 3 and 4. Likewise, single-neutron tunnelling at low energies (the Oppenheimer–Philips mechanism[8]) will not do the job, and an excited resonance state in ^{4}He (ref. 9), because under these conditions the tunnelling time is much longer than the lifetime of the resonant state.

Any enhancement of the fusion rate must then be 'chemical' in origin — the potential barrier between deuterons may change shape substantially, or deuterons may gain some unusual thermal activation.

An increase in the density and effective mass of lattice electrons can cause a decrease of the shielding radius and a corresponding increase in barrier penetrability. In addition, the combined effects of a redistribution of electrons and lattice deformation may produce a local minimum in the potential barrier. Under the right conditions, a quasi-stationary complex can form, and an effect known as resonance transparency may appear[10]. If such a complex has a vibrational frequency with an energy ~ 0.1 eV, the effective collision frequency of deuterons can be greater by as much as a factor of 10^9 over the simple 'lattice gas' value.

A considerable increase in the fusion rate would also come about if a single deuteron were able to acquire ~ 10 keV of energy. In a lattice in equilibrium such an energy transfer would not occur, but perhaps during the destruction of a lattice there might be significant localization of the destruction energy, which could be transferred to absorbed deuterons. A macroscopic version of this effect is found in the electrostatic acceleration of deuterium ions in cracks, which, as noted by Cohen and Davies in their recent News and Views article[11], is apparently responsible for the appearance of neutrons during the shock destruction of D_2O and LiD (refs 1 and 2).

This is then a problem in piconuclear (pressure-induced) reactions rather than in thermonuclear reactions. Common features may apply in the various experiments: high overvoltages and D compression in ref. 3, cold fusion in natural geophysical conditions in ref. 4, and high static pressures and shears in refs 1 and 2.

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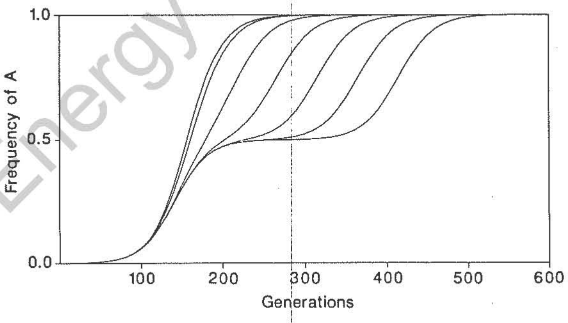
Sex in diploids

SIR—Kirkpatrick and Jenkins[1] suggest that the origin and maintenance of sex in diploids is related to the advantage conferred by genetic segregation. They contend that the segregation advantage gained by facilitating the fixation of advantageous mutations more than compensates for the twofold reproduction advantage of asexual reproduction. We point out here that the segregation advantage they discuss is present even when only a very small proportion of individuals in a population reproduce sexually.

To illustrate this, we have compared the pattern of increase in the frequency of a favourable allele in a population with only sexual reproduction to that in populations where there is a mixture of sexual and asexual reproduction[2]. As an example, the figure shows the change in allelic frequency of advantageous mutation A when there is a 10 per cent selective difference between homozygotes and the heterozygotes are exactly intermediate in fitness. Note first that the initial increase from 0.001 to nearly 0.5 occurs essentially at the same rate for all levels of sexual reproduction. Furthermore, even if there is only a very small amount of sexual reproduction, the continued increase of the advantageous allele from 0.5 to fixation is nearly as fast as that in a completely sexually reproducing population.



The change in frequency of advantageous mutation A for different amounts of sexual reproduction. For the curve on the far right the proportion of sexual reproduction is 10^{-7} (1 in 10 million individuals reproduce sexually). Successive curves to the left are for increasing amounts of sexual reproduction (10^{-6}, 10^{-5}, . . 10^{-1}). The curve for 10 per cent sexual reproduction is indistinguishable from 100 per cent sexual reproduction.

Kirkpatrick and Jenkins show that there is a plateau at a frequency of 0.5 for the heterozygous asexual population while it is waiting for the occurrence of a second mutation. We observe a temporary plateau at an allelic frequency of 0.5 only when the proportion of sexual reproduction is about 10^{-6} or smaller. This plateau, which is generally very short and in the most extreme case in the figure only doubles the time to fixation of the favourable mutant, occurs because the population consists of nearly 100% heterozygotes at the beginning of this period and is waiting for segregation to produce AA homozygotes (which it will at a rate about ¼ times the rate of sexual reproduction). We also find that the relative duration of this plateau is even shorter when the selective difference between the homozygotes is reduced.

A number of species have a small (but not zero) amount of sexual reproduction[2,3]. Perhaps this is a way in which a species can have both the segregation advantage discussed by Kirkpatrick and Jenkins and the twofold advantage of asexuality many others have noted. In other words, we see why some sexual reproduction should be favoured over complete asexual reproduction because of the segregation advantage, but it is not clear why a lot of sexual reproduction should be favoured over a little[4].

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Date: Wed, 8 Nov 89 19:41 GMT +1
From: MORRISON%VXPRIX.decnet.cern.ch@cernvax
Subject: See 11.7 and 11.8 which concern you. hope to send off Thursday.
To: rlg2@yktvmv
X-Vms-To: MINT::"rlg2@yktvmv"

Dear E632 and WA84 Colleague, 1 November 1989.

COLD FUSION NOTE No.20 - Part 2
NEWS FROM LEP

0. A Long Awaited result; the Steve Jones - Moshe Gai Collaboration

00. Fallout from the Closed NSF/EPRI Meeting

5. Visit to Texas A&M

6. Visit to Gainsville,Florida

7. Hot/Cold Fusion Clusters at BNL

8. Ion Implantation and other Research at IPP, Munich

9. Results from the Paul Scherrer Institute, Zurich

10. Discussion of the Tritium Results

11. Other Items
    11.1 Results from Japan
    11.2 Results with Dry Titanium Cells from Culham Lab, UKAEA/Euroatom Fusion
    11.3 Results with Constant Temperature Calorimetery from Brazil
    11.4 Posible Titanium Fracture giving Neutrons at Moscow
    11.5 Results from Frascati/Rome in Gran Sasso
    11.6 Results from Mexico
    11.7 Possible Explanation of Excess Heat
    11.8 Santa Fe Highlights Issued
    11.9 BNL Closed Calorimeter Results Soon

LEP UP-DATE

0) A LONG-AWAITED RESULT; the Steve Jones - Moshe Gai Collaboration

At the Santa Fe meeting, May 24th, Moshe Gai challenged publically Steve Jones to come to Yale and repeat his experiment giving neutron bursts. This is what many had wanted - a believer and a sceptic working together as scientists first. Steve gallantly accepted. At the end of August the experiments were performed at Yale by 7 people from Yale and Kelvin Lynn from BNL plus

Steve.
A non-press Press Release was issued saying they would say nothing until they were ready. Was told in Utah that neutron bursts had been observed but out of respect for their agreement did not repeat it. Now they have issued a preliminary report for the DOE/ERAP panel saying they have found no bursts. Essentially the small fluctuations in the number of neutrons observed both coincided with cosmic ray events.
An abstract of the Abstract is;

In a search for bursts of neutrons from Ti in pressurized D2 gas cells ("dry cells"), no statistically significant deviations from the background were observed for events where 5 or more neutrons are detected over a ten day experiment, including 103 hours of counting with cells on, and 28 hours counting of various backgrounds. Up to four cells were used including some 60 grams (each) of 662 Ti fillings in a pressurised cylinder with 40 to 60 atmospheres of D2 gas.....The samples were cooled to liquid nitrogen temperature and placed in front of the (12) neutron detectors while warming up to
room temperature. Seven cooling cycles (of some 9 hours each) were used for each sample......Two events including 5 detected neutrons were observed in the background runs and two events including 5 and 6 detected neutrons were measured with the cells on. All four events were vetoed by the cosmic ray veto counter and they appear consistent with background events. The 3 sigma upper limit....... is smaller than 27 neutrons over 103 hours from some 200 grams of Ti......

There is a disclaimer (?) saying some further analysis at BYU is in progress so there may be more to come. But the essential point is that the results of Menlove, Jones et al. reported at Santa Fe very soon after the results were obtained, where bursts of neutrons were claimed to be observed after 40 minutes, is not confirmed in a careful experiment with a high quality set of neutron counters.

00) FALLOUT FROM THE CLOSED NSF/EPRI MEETING

The manner in which the engineering section of the National Science Foundation organised the recent cold fusion meeting in Washington is still causing discussion. Invitations were strictly limited and although in the world there are many more groups with negative results than positive results, only about four "sceptics" were invited of whom two refused, whereas most of the remainder(though not all) were "believers" - to use the believers' nomenclature. At the start of the meeting there were some requests to restrict information - some said it was a request for secrecy, that the meeting wass confidential, others that it was only to protect people who were giving preliminary results. This not new but the blanket request is new. Some were surprised there was a press release afterwards as the conference did not come to an agreed conclusion. Now Robert Park of the APS writes that the organisers plan to issue their report just before the DOE panel's long announced date of 15 November for giving their final report. He further writes that the APS President, James Krumhansl, has requested the National Science Board to "examine the cicumstances surrounding NSF involvement" in the NSF/EPRI meeting.

At the start of the meeting Paul Werbos of the NSF said they would like to have a status report on Cold Fusion and a possible programme of basic research. A review talk was given but only published papers were reported - this is a bias since people with positive confirmation are (initially at least) happy to publish while those with null results tend to spend more time checking before saying or admitting that they cannot find a published effect. Nonetheless the papers were more negative than positive. Most papers are not yet published and an up-to-date and balanced account needs to have also submitted papers and other information.

5 VISIT TO TEXAS A&M UNIVERSITY

Dick Arnowitt of the Physics Dept arranged for me to give a lecture on the Status of Cold Fusion and also arranged three meetings with groups obtaining positive results.

5.1 Drs. Packham and Wolf.

Dr Packham who works with Dr. Bockris (absent at the conference in Japan), was initially busy with people from EPRI, but had me shown their cells. There were many small cells, most carrying current, some with stirrers. None seemed to be active(giving excess heat) while I was there. The experiments seemed clean and well-organised. No cell that I saw was completely closed with a catalyst inside. Later Kevin Wolf took me around the Cyclotron Laboratory which is a large organisation with strict entry controls (this was quoted as being important to exclude the possibility of anyone contaminating the tritium samples which were measured there). The neutron measurement lab was in a separate room and looked reliable and showed experience. Was later given several papers;

5.1.1 Tritium measurements

A paper by Packham et al. (J. Electro. Anal. Chem. 270(1989)451-458), is carefully and well-written (and very different from earlier versions). It is found that

1) the production of tritium is erratic in time and in pre-treatment of the cathode
2) the amount of tritium found in the electrolyte varies from 1 E 4 to 5 E 6 dpmml-1 while in a gas sample 5 E 7 was measured.
3) This later version emphasises the many checks that were made including controls with light water(not in earlier version). The tritium measurements were made at 5 different labs and the results were consistent.
4) neutrons were not observed to be produced at the same rate as would be expected from the positive tritium values.

5.1.2 Excess heat production

The paper of Kainthla et al. (Electrochemica Acta 34 (1989) 1315-1318) says that of 10 cells, 3 were observed to give heat bursts, one for 33 hours. The paper is difficult to follow and a complete coherent description is awaited - though the use of a closed calorimeter would simplify things. The effect is basically that the observed heat rise as the power is increased, is more (by less than one degree) than that expected from the calibration curve. There is no discussion of all the possible other effects.

5.1.3 Simultaneous observation of excess heat and tritium production

The amount of tritium observed is much less than what would be expected from the 1.1 or 2 to 5 or 5 Watts as diversely claimed, but Dr. Bockris has just sent me a graph showing simultaneous observation of tritium and excess heat production. From the 11th to 20th September only excess heat was observed but until the 8th October tritium is also observed in both the cell and in the gas. It seems that there is still a discrepancy between the excess heat and the corresponding amount of tritium observed if the excess heat were to come from fusion - but will await the experimenters' paper.

The question of the tritium work is discussed more fully in Section 10.

Was also given a paper "Eight Chemical Explanations of the Fleischmann-Pons Effect" by Kainthla et al. which is a fuller version of the talk that Dr. Bockris gave at Santa Fe. It concludes that "no individual one is sufficient to explain the heat produced. All of them together can only explain heat as much as 3 watts-cm3".

5.1.4 Sparks

Kevin Wolf, who is a serious observer, told me that he had seen sparks coming from a cell while it was in the dark. This is intriguing and should be easy to follow up experimentally. It is perhaps not unexpected as the cathode is under stress and could crack. It would be good to search for coincidences of light flashes with other effects. Similarly it would be useful to follow up the suggestion made by Dick Garwin at Santa Fe to put a microphone against the cathode.

5.2 Drs. Appleby and Murphy

Discussed with Drs. Appleby and Murphy and was given two papers. One describes the measurements reported at Santa Fe and Varenna. The curves look impressive and seem to show a correlation between the excess heat generated and the current in the cells. The derived rate of power generated is in the range 4 to 25 Watts. However what is actually measured is much less, about 40 milliWatts which is safely above the precision claimed of one microWatt - but this is not the same as the background which does not seem to be given. It is curious that as the current is increased from 0.3 to 0.6 to 1.0 A/cm2, the power is roughly steady at 29, 38 and 38 milliWatt - this is explained by deducing that the efficiency decreases from 31.5 to 13.2 to 6.1% resp. This is reminiscent of the positive neutron measurements where the effect seems to be always about three to five times background no matter what the background level is.

The question of the claim to be observing 4 to 25 Watts/cm3 could easily be solved by making a bigger cell with a one cm3 cathode and actually measuring 25 Watts and not 40 milliWatts e.g. by boiling some water.

Was given Tables of the latest results which showed that between the 11 and 15 May (before Santa Fe) excess heat of 13 to 23 Watts/cm3 were obtained consistently and between 27 and 31 May (after Santa Fe) further measurements were carried out with light Hydrogen instead of Deuterium and no excess heat was observed, with values ranging from -3.28 to +0.01 Watts/cm3 (this could be considered a measurement of the background). In both cases the electrode was previously charged for two days at 0.06 A/cm2 - this is interesting as it answers those who claim that to obtain positive effects it is necessary to charge for long periods (months), and this is consistent with measurements of the time to load the cathode with deuterium of about half a day. Further the positive results of 20 Watts/cm3 were obtained with cell currents of 0.098 A/cm2 - this answers those who claim that effects can only be obtained with very large currents.

The second paper was interesting for the comments it makes on Pons and Fleischmann's work and a possible influence of lithium. The essential result was that when the electrolyte was changed and Sodium hydroxide was substituted for Lithium hydroxide, then the excess heat ceased. Since Lithium can penetrate into the palladium catalyst but very slowly, it is suggested that "the results show that excess enthalpy production requires the presence of lithium in the electrolyte, indifferently the 6Li or 7Li isotopes. This suggests that a superficial chemical process underlies the effect, although it does not mean that the excess heat is of chemical origin." This is in contradiction to Fleischmann and Pons who said it was a volume effect though "their data scarcely suggest this, since at 0.008 A/cm2 changing the electrode volume by a factor of 16 increased the excess heat flux by only 28%."

As far as I can make out no new work has been done since about the end of May and Drs Appleby and Murphy explained that this was caused by an absence of funding. This is surprising in view of the nature of their results and in view of the fact that they are claiming a much higher reproducibility rate than anyone else - about 80% (though it did not work when the DOE Panel came for a very brief visit). In their first paper (quoted "Nature, in press"), it is written that the work was supported by EPRI.

Since the work is intriguing, one would be inclined to believe it if it were repeated with reasonably sized cells which really gave 20 Watts/cm3.

5.3 Lecture on the Status of Cold Fusion.

Gave the same lecture on the Status as at Utah. Again it was reasonably well received by a smaller audience. However during the questions and comments had some powerful remarks by a non-academic person saying that it was very difficult to get positive results and everyone who did it had to work very hard, but to find nothing was very easy and implying that people who did not find anything did not work hard. This rather shocked me and I replied that there were two things;

1) I knew many of the people who had obtained null results and who had placed stringent upper limits on the effects, and they had worked very hard - quoted

the case of David Williams' group at Harwell who for three months never had a day off
2) Found his remark insulting to people who had not found any effect, and suggested he might like to withdraw his comment.

Looking at him I realised he would not reconsider his comment, so to avoid trouble for my hosts, switched to the next questioner.

It would be comforting to think that these non-scientific slurs were confined to non-academic people, but found the following in a paper "a principal difficulty has been that many electrochemically inexperienced investigators have tried but failed, to reproduce the alleged heat" - not too subtly suggesting the reason they failed was because they were inexperienced. But people like Drs. Williams, and Kreysa to mention only two who are friends of Martin Fleischmann, are experienced electrochemically, plus Drs. Lewis, Petitjean and others and have also tried and "failed" to reproduce excess heat but have given tight upper limits on the effect which are less than the phenomena claimed.

6. VISIT TO GAINSVILLE, FLORIDA

In Gainsville there are two groups in the same building working on Cold Fusion. The Downstairs group of Profs Schoessow and Wethington have reported that they have observed heat, neutrons and tritium and the Upstairs group of 14 people of different skills, led by Dr. Muga who have found no effects.

Working part-time, Profs Schoessow and Wethington have essentially built their apparatus themselves, having an average of 74, this is a fine achievement. It consists essentially of a large container with two cells in it and water flowing through. The temperature of the water is measured entering and leaving each cell, and the rate of water flow is measured with a graduated container and a stopwatch - simple but effective. Their initial tritium measurements were controversial because of doubts about the purity of the D2O added. Their neutron counting system is very simple and would be quite adequate if they were to observe fusion producing say 0.1 Watts for then there would be 1 E 11 neutrons per second. However if the neutrons to be measured were only 10 or 100 times background, then from experience, such a simple system is not enough for the precise measurement of neutrons and for distinguishing them from backgrounds from radon, radioactive decay in plaster, concrete etc, and from gammas. The measurement of neutrons is very difficult and takes, in general lots of experience (will try and distribute a note later about how not to measure neutrons). The gamma ray detector looked safer and could give reliable results after controls. The calorimetry looked delightfully straightforward. A first cell was found to give 60% excess heat with D2O but only 4% with H2O. During my visit one cell had been running for 27 days and the other for 16 days and were giving about 40% excess heat. The current was generally about 100 to 200 mA but had been varied between 30 mA and 1 Amp.

Mentioned to Glen Schoessow that I was worried the calibration had not been done and suggested a simple heater be inserted in the cell and the change in water temperature measured as the power of the heater was varied. When I arrived in Dallas three days later, Glen's son and daughter-in-law met me and showed me Dallas. At dinner Tom said 92% - his father had asked him to tell me 92%. When I phoned Glen he explained that the next day he had stopped one cell and had inserted a heater he had made and had done the calibration finding that he was getting 92% of the heat in. This meant that he was losing 8% to the surroundings. As this was much less than than the 40% excess, it confirmed the effect. This was so over the range 5 to 20 Watts but the loss rose to 12 % for 25 Watts input.

The apparatus of the Upstairs group was much larger and more conventional apart from one few piece that I liked. This was a cell with the electrodes far apart (10 cm) and a burette over each. The amount of D2 and O2 gas was collected over each electrode. It was found that there was much less D2 as it was being absorbed by the palladium. This allowed them to measure the amount of the loading of Deuterium into the Pd. It was observed that the Pd loading reached a plateau after a some hours (the exact numbers have not been fully analysed yet).

At lunch and after my lecture the Upstairs and Downstairs groups talked together.

7. HOT/COLD FUSION BY CLUSTERS AT BNL

The New York Times had a large article saying that fusion had been observed at Brookhaven National Lab and it related this to Cold Fusion, Fleischmann and Pons and the possibility of power generation. What had happened was that Beuhler et al. at BNL had developed a new technique of accelerating clusters of D2O molecules by giving the cluster a charge, and making the beam impinge on a target of Titanium loaded with deuterium. They observed protons of 3 MeV and a peak at 1 MeV which they ascribed to tritons. This is strong indication of the occurence of the fusion reaction d + d ---> t + p + 4 MeV. The paper was published in PRL 63(1989)1292. It was presented at a meeting in Florida where it was considered of unusual interest, released and written up by the NYT. Brookhaven has been careful not to say it is Cold Fusion for people consider it more likely to be Hot Fusion - maybe Lukewarm Fusion. As the numbers of protons produced with great effort and energy, are very few, it is clearly not a potential new energy source.

In the PRL paper only proton and triton production were shown which may have encouraged those who believe that it is possible to obtain large amounts of tritium without at the same time producing equal amounts of neutrons. However the reason that the helium three of 0.8 MeV was not observed initially was that it was drowned in the background. Several groups are now studying the phenomenon at BNL - the background has been reduced and the 3He peak is clearly seen - this from the reaction d + d ---> 3He + n. The rate of 3He production is of the same order of magnitude as the tritons, not eight orders of magnitude less as some believers in the tritium results have suggested. This question of the tritium results is discussed more fully in Section 10.

The clusters vary in size from 25 to 1300 molecules of D2O. They are singly charged and accelerated to up to 325 keV.
The mechanism is not yet established. Steve Koonin has written a paper giving three models. The standard thick target and the thick target yield for deuterons knocked-on by oxygen ions, are both many orders of magnitude too small. The thermonuclear yield from an isoergically expanding gas of atoms agrees with the results for clusters of 25 to 50 molecules but is many orders of magnitude too low for clusters above 200 molecules. Dick Garwin told me that considerations of double knock-ons gives a velocity to the deuterons something like three times the cluster speed and accounts for the very gradual variation of yield with energy; but the varaiation with cluster size cannot be understood this way. Steve is having more complete calculations done. See below in Section 8 for related work.

8. ION IMPLANTATION AND COLD FUSION TESTS AT MPI, MUNICH

The Institute for Plasma Physics at the Max Planck Institute, Garching, Munich has reported three attempts to reproduce Cold Fusion;

1) Electrochemical Cold Fusion tests following Fleischmann and Pons,
   Wurden et al., Report IPPIII/149, July 1989
2) Search for Fusion Reactions between Deuterium Atoms implanted into Titanium. Behrisch et al. Nucl. Fusion 29(1989)1187
3) Fusion reactions observed during the actual implantation of Deuterium ions into Titanium. Roth et al., submitted for publication.

The summary letter from Dr. V. Dose says

"The results of our attempts are entirely negative. The electrolytic approach did not reveal any of the phenomena reported by Fleischmann and Pons, in particular no excess heat was observed" "in conclusion the IPP group terminates Cold Fusion research until those who find positive indications, home in on reproducible experiments."

There is a hint of suppressed anger at the waste caused. Robert Park of the APS writes this week that "estimates of the total expenditure on cold fusion research start at $50 million." Wonder if he is aware of these MPI experiments, and others.

However the IPP work did yield some useful results. D+ ions of from 300 to 6000 electron-volts (not keV as said in Part 1) were implanted into a 3 micron thick foil of Ti at room temperature and at 140 Kelvin. At room temperature there were 1.8 D per Ti atom while at 140K there were 3 D atoms per Ti atom (values of up to 5 have been obtained). Protons were measured showing that the d + d ---> t + p reaction occurs. The background was very low, 2 E -5 counts per second. The cross section measurements were at the lowest energy for this important reaction (Dick Garwin mentioned that he had some responsibility for some of the earlier measurements by Arnold et al.) It is interesting to note that the results follow the Gamow curve indicating that nothing anomalous occurs at very low energy.

9. RESULTS from the PAUL SCHERRER INSTITUTE, ZURICH

The groups that had worked most on Cold Fusion research before 1989 were those of Steve Jones in the USA(at Los Alamos) and of Claude Petitjean at the Paul Scherrer Institute (formally called SIN, but sin has now been ended in Switzerland - perhaps). Both groups were able to obtain fusion by capturing muons over 100 times before the muon decayed. So it was natural for the PSI to mount a major effort to study the new form of Cold Fusion as observed in Utah. Further the group also worked on electrochemical energy storage therefore were doubly experienced in the subject. The paper, (PSI report PR-89-17 and submitted to Chimia) is a mature one giving references going back to 1869. An electrochemical cell with D2O and an identical cell with H2O were used for calorimetry studies and neutron and gamma detectors were positioned near the cells. The cathodes were also examined for 3He and 4He by mass spectroscopic analysis. Four conditions of operation were made with different cathodes and currents between 120 and 500 mA/cm2.

The calorimetry was carefully done keeping a constant temperature which greatly reduces the corrections and calibrations needed. The calculations and operating conditions(including temperatures) are fully given. The recombination rate of the D2 and O2 gases was found to be less than 3%. The overall error was estimated to be +/- 100 milliWatts. During two months no excess heat was observed. H-D fusion was also tried but again no excess heat was observed.

Gravimetric H and D loading measurements in Pd gave values of 0.85 to 0.95.

The neutron measurements were made with a NE213 liquid scintillator and found no evidence of 2.45 MeV neutrons. The background was 0.04 n/s. The upper limit on neutron production was 0.14 n/s or < 1 E -13 Watts and is below the positive observations of Jones et al.

Gammas were searched for using a BGO detector and an upper limit of 0.1 gammas per sec. was found.

No excess 3He was observed above that expected from the decay of tritium. Although 4He measurements are difficult because of contamination, an upper limit six orders of magnitude lower than that reported by Fleischmann and Pons was obtained.

Tritium measurements were made and found consistent with an isotopic separation factor of 2 and with the measurements of Comninellis et al. at the EPFL in Lausanne. There was no indiction of the increase of tritium of many orders of magnitude as reported at Texas A&M.

Claude has sent the published version in Chimia 43(1989)262-268.

10. DISCUSSION OF TRITIUM RESULTS

Am often asked how do I account for the tritium results? The questioner is normally referring to the positive results found at Texas A&M, Los Alamos, and Case Western Reserve. He is not referring to the null results obtained at these same three places, since some cells do not give an effect. Nor is he referring to the null results at PSI, and five other labs.

The answer is that one should look at all the results and knowledge. The proponents say that observation of tritium is an indication that fusion has taken place. Let us assume that it is so and look at the consequences;

1) There is a great weight of experimental evidence that neutrons are not produced in Cold Fusion at anything near the rate claimed for tritium and in fact most experiments find no neutrons at all. The response of believers is that the neutron production is reduced by a factor of E-8.

The reactions

$$d + d \longrightarrow t + p \qquad (1)$$

$$d + d \longrightarrow {}^3He + n \qquad (2)$$

should have the same cross section by isospin and there is a great deal of evidence that this is so. The BNL Cluster fusion work is one of the most recent results; the IPP, Munich result shows there is nothing anomalous at very low energy in reaction (1). Dr. Teller emphasised this at the NSF/EPRI meeting.

Thus it would take a miracle to reconcile the tritium and neutron results.

2) If tritium is produced in reaction (1), then they should have one MeV of energy. These would produce the well-known reaction

$$t + d \longrightarrow {}^4He + n \qquad (3)$$

and give large quantities of 14 MeV neutrons. Such energetic neutrons would be very easy to observe, but have not been found.

It would take a miracle to have 1 MeV tritium and not 14 MeV neutrons.

3) If the tritium is produced in reaction (1), then an equal number of protons of 3.02 MeV energy should be produced. These will excite the even-even isotopes of Palladium by 0+ to 2+ transitions, and these will de-excite by the emission of gammas of betwen 0.37 and 0.56 MeV. This was confirmed experimentally by Salamon et al. who with the help of Dr. Schier of the University of Lowell, bombarded Palladium with 3.02 MeV protons and observed the expected peaks at 0.37, 0.43 and 0.51 Mev at their expected strengths. But no such gamma rays have been found e.g. when Mike Salamon operated for five weeks, his counter under the table where Dr. Pons had four cells running.

Thus if we wish to believe that tritium is produced in the fusion reaction (1), we need three miracles in addition.

Note that the irreproducibility of the results and the inconsistency of most tritium rates with the amount of excess heat claimed, are not included in the number of unusual occurrences.

11 OTHER ITEMS

11.1 Results from Japan

Positive results on Cold Fusion were slow in appearing in Japan. The first announcement by Drs Mizuno and Akimoto assistant professors at Hokkaido University of the observation of neutrons, was apparently delayed as previously a professor there had already announced that after his tests cold fusion did not exist. The two younger researchers may have wished to avoid bring shame on their colleague. Their experiment was carried out in a shielded underground lab. The neutron rate was 10 times the background of about 0.3 n/s. It is said that the background was measured for 7 days before the experiment began. This is poor technique as the background is often found to fluctuate with time. It is to be hoped that later measurements were made with more than one counter measuring simultaneously.

Dr. Takahashi et al. of the University of Osaka Engineering Dept. detected neutrons above background and 2 to 4 times more than Jones et al. They switched the electric current between 0.8 and 0.4 Amps every 2.25 minutes. Fast neutrons of 2.4 MeV were observed.

Prof. Oyama of Tokyo University of Agriculture and Technology found tritium but one week later the Japan Atomic Energy Research Institute working with him, found almost no neutrons or tritium. However this joint team announced 31 July that they had found tritium and considerable heat.

At the 31 July meeting there were reports of 10 teams obtaining positive results. Prof. Bockris told me that at the Kyoto meeting at the end of

September, there were 21 papers of which 8 gave positive results.

Do not have recent information and that given above may be out of date by now, but it does give a flavour of the situation. Everyone says there is a great deal of work in Industry whose results are unknown.

11.2 Results with Dry Titanium Cells from Culham Lab, UKAEA/Euratom Fusion

G. M McCracken et al.

report on a major series of tests of the results reported by Frascati on the observation of neutrons when titanium samples are loaded with deuterium, cooled to liquid nitrogen temperature and then warmed up. The experiments were prepared at the Culham laboratory of the United Kingdom Atomic Energy Authority and the neutrons were searched for in three places (1) at Culham with a single simple BF3 counter

(2) At the Joint European Torus, JET, tokamak where there is a system of six 3He counters which is in regular use and whose characteristics are well known.

(3) At the UKAEA Harwell lab where 32 detectors with 3He were used.

No neutrons were detected with the ten samples used. They give a limit of 0.14 n/s which is four orders of magnitude lower than reported by De Ninno et al.

If neutrons were emitted in brief bursts (< 0.1 s), they would have to be less than 30 neutrons otherwise they would have been detected.

A possible explanation of the early results from Frascati where a single BF3 counter was used and which Frascati cannot repeat since), is given. "Our experience in making measurements of low fluxes of neutrons has indicated that proportional counters of both the BF3 and 3He type are subject to random fluctuations in count rate. These fluctuations can in some cases exceed the average background count by two orders of magnitude. Thus clearly a number of independent counters are necessary". The point being that occasionally one counter will give counts but the others do not which shows the effect is not caused by neutrons.

For connaisseurs, the description of the preparation of the Ti is interesting The Ti sample is first heated to 460 to 900K to remove impurities. It is then put in a closed vessel and D2 gas added - the pressure drops as the Deuterium is absorbed and this allows the loading to be estimated. Also as the absorption gives out heat (50kJ/mole) the temperature rises by a substantial amount. Normally a loading of D/Ti = 2 was achieved but in one case, where the Ti was initially baked at 460K, there was no absorption or temperature rise which was attributed to a layer of oxide on the Ti surface blocking the Deuterium uptake. When the Ti samples were heated to 900 K, the oxide layer was removed and the results were reproducible.

11.3 RESULTS FROM CONSTANT TEMPERATURE CALORIMETRY IN BRAZIL

Prof. Ed Wrenn

of Utah told me had visited the lab. of Mechanical Properties at the Centro de Tecnologia, Rio (which I am told is perhaps the best engineering university in Latin America). They have a well-instrumented cell with a Pd cathode and can measure heat excursions down to 12 mWatts. Over a period of 3000 hours, they have not found any excursions. Neutrons are also being looked for.

11.4 Possible Titanium Fracture giving Neutrons at Moscow

At the Institute

for Physical Chemistry, Moscow, B. V Derjaguin report in Nature 341(1989)492 some first results of an attempt to observe neutrons from Titanium which they put under stress by placing Ti chips with deuterated materials and steel balls in a vibromill operated at 50 Hertz with an amplitude of 5 mm. They claim to have observed neutrons but there are only 29 counts spread over 31 channels so the evidence is rather marginal. The highest rate claimed is 0.41 +/- 0.14 counts/s. As each test seemed to have lasted less than an hour, it would be good to await their final results when they have run for a week at least.

11.5 Results from Frascati/Rome in Gran Sasso

F. Celani et al. from Frascati and Rome reported at the Varenna Conference on their results with an electrolytic cell with Pd and Ti electrodes carried out in the Gran Sasso tunnel as the background is very low. For neutrons (after further shielding with lead bricks against gammas) it was 1 E-6 n.cm2/s which is 1000 times less than at sea level. For gammas it is more difficult and was only 10 times less.The Pd electrodes used were 0.2, 2, 4, and 8 mm diameter and the Ti bars were of 1 and of 5 mm side. There was a NaI(Tl) gamma detector, two 3He counters for neutrons and fast plastic scintillator to detect charged particles.

Two emission events were observed;

1) a "shot" signal(lasting < 0.6 s) followed about 40 minutes later by some bursts of signals (during 4 minutes) in almost all detectors on May 2 at 23.30. The 8 mm diameter rod had begun to be charged only 30 minutes previously - this time is so short that the loading of Deuterium into the Pd must have been very light (the charging current was 66 mA/cm2) so that it is inconsistent with most other accounts of Cold Fusion phenomenon and another explanation may be preferred.
2) The Ti electrode was stressed by several thermal cycles (from -196 to +200 C) and cathodic and anodic currents of 3A, and mechanical stress. Four bursts were observed with the gamma detector. This on 1st May at 13.30

In both cases no charged particle emission was observed.

These two results both had good statistics.

The question is if they had any connection with cold fusion since they were observed on May 1 and 2 but when reported at Varenna four and a half months later, no other phenomena of a similar exciting nature were claimed.

Note this is the Laboratori Nazionali di Frascati and not the National Laboratory for Alternative Energies which reported neutrons from dry Ti.

11.6 Results from Mexcio

Have received a paper entitled "Experimentos de Fusion Fria en el IMP" by J. M. Malo et al. The Instituto Mexicano del Petroles has made measurements of gammas and tritium. A gamma ray spectrum of the background shows peaks at 1.764 and 2.204 MeV from 204Bi and 2.614 MeV from 208Tl. With the cell running, these same peaks are seen plus a small peak above the 2.204 peak which is labelled

p + n ---> d + gamma   2.224MeV

Fine except that when I looked at it the peak looked too high and on calibrating with the background peak would estimate it to be at about 2.425 MeV. The authors also show some evidence in favour of tritium production and say that their calorimetric work is very difficult to control (they have to employ Newton's Law of Cooling) but conclude that the work should be considered as preliminary and should NOT be taken as conclusive evidence. Thus this was a fair internal document and it illustrates the potential danger of letting such a document become external.

11.7 Possible Explanation of Excess Heat

At the Santa Fe conference, Dick Garwin in his curtailed intervention, suggested that a possible source of excess heat was that the input was assumed to be IV (more exactly I V - 1.54) which assumed that the current was steady whereas if there was a fluctuating component, then the heating being proportional to I2, would be greater but not taken into account. This could be caused by a feed-back occurring. He tells me that this has been found by A Bruggman et al. at Mol in Belgium who observed large "excess heat" from an oscillation of the feedback circuit controlling the electrolysis current.

Thus all groups finding excess heat effects should clearly check for this possibility. However while this could be an explanation in some cases, it is unlikely to explain all for many experimenters have done substantial running with H2O instead of D2O and do not find excess heat effects. So where the number of hours run with light water is comparable with that with heavy water, (not always the case) then such effects are very unlikely. Still people should be aware of this possibility and check that such effects do not cause heat excursions.

11.8 Santa Fe Highlights Issued.

People attending the Santa Fe conference wondered why $400 was charged for so little and there were many jokes about the first time a profit had made out of Cold Fusion. Los Alamos has now issued their official account of the conference and the doubts continue - it is only the Highlights and only the Abstracts - nicely produced but only 26 lightly covered pages - not for example containing the important contribution of Dick Garwin. Doubt if the main summary speaker will like his account, for on speaking to him afterwards I found that he liked Irving Langmuir's article on Pathological Science - but then the organisers kept him as Summary speaker even when he warned them that he would have to miss half the meeting! There are many excellent people at Los Alamos but the relationship between Los Alamos and Cold Fusion is very different from that of the other major laboratories.

11.9 BNL

NEWS FROM LEP

On 7 November the LEP Committee had their open session. Emilio Picasso described possible future plans aiming for higher energy, higher luminosity and polarisation.

After a slow restart, the last week of LEP before the 6 November shutdown, was very productive with 50% of the time spent in Coast. The highest initial luminosity was 4.9 E 30 and the highest beam currents for physics were 1.6 and 2.65 mA.

There will be four days on Machine Development then the official Inauguration of LEP on Monday 13 November.

The total number of Z0's obtained is about 40 000 but the criteria used by the experiments varied, some giving the total number and some the number after all selections and cuts had been made to give a sample which they would use for physics. It is hoped to increase this number appreciably in the 26 days scheduled for Physics before Christmas

We have been running tests of our scintillating fibres for WA84 in Omega using a pion beam from the SPS accelerator while LEP was running. It was amazing to us that we were virtually unaware that the SPS was also being used to accelerate electrons peroidically at the same time. To be able to accelerate protons, electrons and positrons in the SPS with minimal interference is a real achievement.

Douglas R. O. Morrison

Upper bounds on 'cold fusion' in electrolytic cells

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Experiments using three different calorimeter designs and high-efficiency neutron and γ-ray detection on a wide range of materials fail to sustain the recent claims of cold fusion made by Fleischmann *et al.*[1] and Jones *et al.*[2]. Spurious effects which, undetected, could have led to claims of cold fusion, include noise from neutron counters, cosmic-ray background variations, calibration errors in simple calorimeters and variable electrolytic enrichment of tritium.

RECENT publications[1,2] reporting electrochemically induced nuclear fusion, at room temperature, have aroused great interest. The signatures reported are excess heat output, neutron emission and tritium generation from cells with palladium cathodes[1], and neutron emission alone at a much lower level from cells with titanium cathodes[2]. Conventional nuclear physics predicts that fusion between light nuclei requires either very high temperature (as in a tokamak) or unusually close proximity of the two nuclei (as in muon-catalysed fusion). The calculated fusion rate at the internuclear separation in the deuterium molecule (0.74 Å) is $\sim 3\times10^{-64}\ \mathrm{s}^{-1}$ (ref. 3), so the rates reported in ref. 2 ($\sim 10^{-23}$ d-d-pair^{-1} s^{-1}; d is deuteron) are not easy to understand in the context of the known interstititial-site separations (~3 Å), even more so when it is realized that this rate is a severe underestimate because of incomplete deuterium loading in titanium (see Materials characterization section). The reaction rates reported in ref. 1 are even more difficult to understand[4], and the existence, despite strong arguments to the contrary (ref. 5, for example), of an unknown mechanism, which results both in an extraordinary enhancement of the reaction rate and a suppression of the normal nuclear-reaction channels, has been postulated.

In the first reports of the electrolytic cold fusion effect it was stated that the effect is not consistently reproducible, and that it both takes some time to appear and that it may subsequently disappear. Because electrochemical phenomena can be sensitively affected by the state of the surface, some irreproducibility is not, in itself, surprising, and other recent reports (refs 6–8, for example) give well documented accounts of failures as well as successes (R. A. Huggins and A. J. Appleby, Workshop on Cold Fusion, Santa Fe, May 1989) in observing the claimed effects. The clear lack of reproducibility necessitates significant replication, with controls at least equal in number to the number of tests, if positive results are to be viewed with confidence, and exploration of many different, well characterized, material and electrolyte combinations. Particularly, the timescales and achievable concentrations for electrolytic loading of deuterium into palladium and titanium, the quantities of hydrogen 'impurity', and the species detectable by surface analysis of used cathodes ought to be determined.

Calorimetry

We used three types of calorimeter. First, we built calorimeters of size and design similar to those used by Fleischmann *et al.* (ref. 1 and M. Fleischmann, personal communication) (Fig. 1). We found these to be inaccurate instruments with some very subtle sources of error which it is necessary to appreciate and analyse in detail. We used sixteen such cells, containing different-size cathodes (1-, 2-, 4- or 6-mm Pd rods) and different electrolytes (0.1 M LiOD, 0.1 M LiOH, 0.1 M NaOD or 0.1 M NaOH). Figure 2*a,b* shows the results obtained for a typical cell. An immediately evident characteristic is the sloping baseline, with the sawtooth pattern a consequence of the regular refilling of the cell. The baseline slope can be quantitatively accounted for by a variation of the calibration constant, *k*, with the level of liquid in the cell. This is a result of radiative losses through the vacuum jacket[9] and conduction up the glass inner wall of the cell[10] (see Fig. 2 legend). A consequence of the sloping baseline is that any calibration is only valid for a particular liquid level. We regularly refilled these cells to a reference level at which the calibration had been performed. We calibrated the cells with the heaters before the electrolysis was started and again much later in the run, during the electrolysis, as is evident in Fig. 2*a* (see Fig. 1 legend for details of the calibration procedure). There was no statistically significant difference between the different calibration sets, so all data were combined.

Figure 2*c* shows results for two of the calorimeters at the calibrated liquid level, in the form of percentage excess of apparent output power over the Joule input power. There was apparently an endothermic period at the beginning of the run, whose duration increased with cathode diameter. We speculate that this was due in part to poor stirring of the solution during hydrogen uptake by the cathode which caused a temperature gradient in the cell (gas is not at first evolved at the cathode and the larger diameter cathodes were shorter, to keep a constant surface area). An analogous effect at the start of the electrolysis was reported by Lewis *et al.*[8], who showed that the apparent heating coefficient of a similar cell varied during the first part of a run. Because of this effect, we excluded the first 10,000 minutes of data from each cell when calculating the statistics. Table 1*a* shows the mean absolute power deviation for each cell and the standard deviation in this value. The standard deviation for all of the H_2O cells was not significantly different from that of the D_2O cells (*F* test, 1% level) so that the data from all of the cells can be used to estimate the error: $\sigma = 0.048$ W (that is ±5–10%). A more detailed analysis of this error[10] showed it to be largely determined by the variability in the liquid level after refilling the cell, but also with a contribution from unquantified variations in the heat loss by conduction up the calorimeter wall to the air above the water bath. Compared with these errors, effects that resulted from temperature gradients inside the cells were minor. The design used here varied from that in ref. 1 in that the glass sleeve that supported the calorimeter and which was in direct contact with the inner wall, provided a large area of thermal contact with the water bath, and thus reduced the effect of the ambient temperature variations.

As we expected, occasional points from both H_2O and D_2O cells lay outside the 'control limits' of $\pm 2\sigma$ (Fig. 2*c*). No points lay outside $\pm 3\sigma$. No cell showed two or more consecutive points outside $\pm 2\sigma$, and the number of points lying above the control limit in the D_2O-cell experiments was no different from that in the H_2O-cell experiments. Therefore, we conclude that, within the experimental error, there was no significant anomaly in the behaviour of the D_2O cells compared with that of the H_2O cells. If, however, the mean power deviation (Table 1*a*) for all eight of the D_2O cells ($+9.91 \pm 0.47\%$ or 0.021 ± 0.004 W; mean Joule input 0.96 W) is tested against that for all eight of the H_2O cells ($-2.46 \pm 0.72\%$ or -0.018 ± 0.004 W, mean Joule input 0.81 W for LiOH cells and 0.61 W for NaOH cells), it is clearly highly significant. Because no sequence of individual points lay outside the control limits established above, we suspect that there is another, unknown source of error that scales with the input power. This, of course, could be the postulated 'fusion effect', but the magnitude of the effect is commensurate with the errors. The only reliable way of checking this is to construct calorimeters that are free of major sources of errors, and in particular, do not have a sloping baseline. We therefore also used both isothermal and steady state (heat flow) calorimeters which satisfy this criterion.

We have also analysed in detail the slope of the output curve[10] to look for momentary power pulses on timescales shorter than the interval between calibrated points. If the Joule input remains constant then this slope should not vary. Any sudden or momentary extra power input, q_c, would change the slope by approximately $(k_{sb} + k_{c,0})q_c/M_0$ (M_0 is the water equivalent of the calorimeter, other symbols defined in Fig. 2 legend). The number of significant deviations from the mean slope was found to be roughly the same for both the H_2O and the D_2O cells. For the whole data set, the largest power excursion for a D_2O cell (6-mm

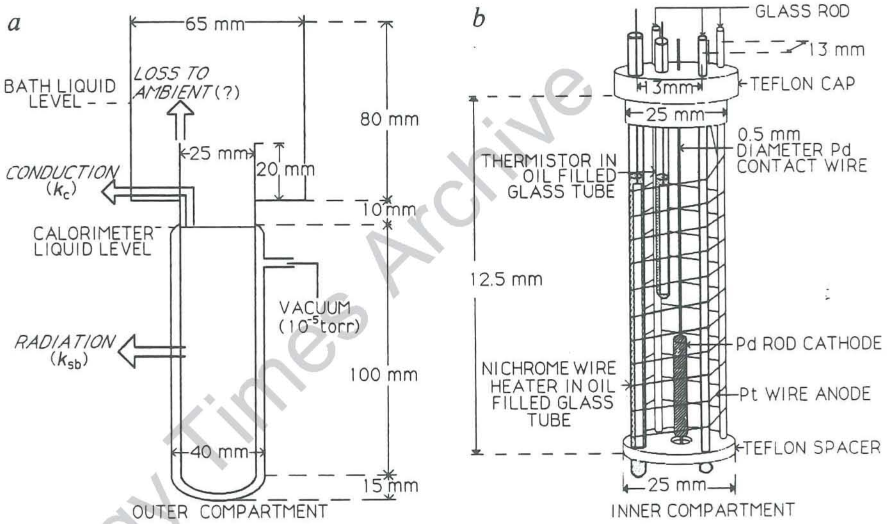


FIG. 1 Schematic diagram of the FPH-type heat-flow calorimeter used here. Heat flow paths are indicated here and discussed further in Fig. 2 legend.

METHODS. As well as the Fleischmann, Pons and Hawkins (FPH) type, we used[10] two other calorimeter types—an improved heat-flow calorimeter (IHF) and an isothermal calorimeter. The IHF calorimeters differed from the FPH type in three important ways. First, they were larger: a 500-ml-capacity cylindrical vessel constituted the cell, with anode–cathode spacing ~2 cm. Second, the electrolysis vessel was inserted, using a film of oil for thermal contact, into a tightly fitting aluminium can which was itself packed around with insulating material and placed in a Dewar flask. Whereas the temperature of the cell contents in the FPH calorimeter was determined using a glass-clad thermistor immersed in the cell itself, in the IHF design the temperature of the aluminium can was measured: the can defined an isothermal surface for conduction of heat away from the cell and its use as the temperature measurement surface eliminated the sloping-baseline problem of the FPH design. Third, the space within the Dewar flask above the electrolysis vessel was filled with a polystyrene cap extending well above the Dewar flask, with the aim of significantly reducing unquantified heat losses to the atmosphere. The two types of heat-flow calorimeter were operated in water baths held at a constant temperature of 20 °C (±0.08 °C). The tops of the water baths were covered with a polystyrene lid. In both cases the cells comprised a spirally wound Pt wire anode (0.25 mm) and a central Pd cathode (Johnson Matthey). For the FPH cells we prepared the different electrolytes using either conductivity water (H_2O cells) or slightly tritiated D_2O (specific activity 13 kBq ml^{-1}) of isotopic purity initially >99.9%. We used 0.1M LiOD (D_2O from Aldrich, measured >99.9% initial isotopic purity) as the electrolyte in the IHF calorimeters. The Pt and Pd contact wire was shrouded in glass tubing in the IHF cells to prevent any possible catalytic recombination of the electrolysis products. In later experiments with the FPH cells we also used screened electrode contacts, but this had no effect on the results obtained. The IHF cells contained a Pd wire electrode insulated to the very tip, which was positioned about three-quarters of the way up the cell. This was used to define the internal liquid level during filling, or refilling, of the cell. The larger volume of electrolyte in the IHF cells (300 ml) meant that refilling was required only occasionally, but the large thermal mass meant that the response was slow (time constant ~12 h): thus they were not sensitive to small bursts of heat. We calibrated the heat-flow calorimeters using a nichrome wire heater placed in an oil-filled glass tube which was in contact with the cell contents. The calibration procedure involved operating the heater, either without electrolytic current, or with a current significantly less (0.2–0.4 times) than the normal electrolysis current, until a steady-state temperature was attained. With the FPH cells, use of a lower electrolysis current ensured continued stirring while diminishing the errors arising from the baseline drift; before each individual calibration the cells were refilled. An empirical calibration curve was thus obtained by fitting the observed thermistor resistance, R, to a range of applied powers, P, using the equation $P = a - b(\log R) + c(\log R)^2$, where a, b and c are the fitted parameters. The standard error of estimate on the fitted curve varied from 10 to as much as 70 mW with the FPH cells, largely reflecting the error in extrapolation to the reference level. Neutron counting was performed in conjunction with the operation of the FPH cells, using two independent banks of counters mounted above the cells, on top of the water-bath cover[10]. Detection sensitivity above background was 3 event s^{-1} in the cell. We never observed a signal above background. The isothermal calorimeter (J. A. Mason, R. W. Wilde, J. C. Vickery, B. W. Hooton and G. M. Wells, Proc. 29th A. Meet. Inst. Nuclear Materials Management, Las Vegas, Nevada, June 1988) comprises three concentric aluminium cylinders each separated by a heat transfer medium with a relatively low thermal conductivity. The cylinder temperatures are maintained by electrical heaters wound as helical coils around each cylinder. Temperature control is achieved by resistance thermometers on each cylinder which are used in conjunction with classical control software and the cylinder heaters. The rate of thermal energy evolution in the measurement chamber (12.5-cm diameter, 26 cm high) is determined by measuring precisely the electrical power supplied to the chamber. We operated the calorimeter at a measurement-chamber temperature of 42 ± 0.001 °C. The measurement-chamber power resolution is <5 mW for an operating power of 20 W. The electrolytic cell contained ~1 l of 0.1M LiOD. The cathode was contained in a perforated glass canopy to prevent the evolved gases from mixing inside the measurement chamber. The anode was a Pt foil cylinder 3 cm high and 12 cm in diameter surrounding the cathode. The cell was thermally coupled to the calorimeter measurement chamber by conducting oil. Measurements using Pt cathodes and also using nichrome wire heaters showed that there was a small systematic error in the calorimeter, an apparent power excess that varied linearly with the input power up to 100 mW for an input of 15 W. A linear fit to these measurements (12 points, standard error of estimate 8.8 mW) was therefore used to apply a correction to the apparent excess measured for the Pd cathodes. The measured output power of both the isothermal and IHF cells was corrected for the power loss that is due to evaporation of the electrolyte, assuming that the electrolysis gases were saturated in water vapour as they passed out of the cell (25.0 mW A^{-1} at 42 °C): $q_v = (p_v/p_0)(1.5)\Delta H_v I/(2F)$ where q_v is the power loss, p_v denotes the saturation vapour pressure and ΔH_v the latent heat of evaporation of the electrolyte, p_0 is the atmospheric pressure, F is the Faraday constant and I is the current (assumes ideal gases).

TABLE 1 Calorimetry results

(*a*) FPH Calorimeters: current 300 mA, current density 80–110 mA cm^{-2}, 0.1 M LiOH, NaOH, LiOD, NaOD

Pd[a]			H_2O Cells		D_2O Cells[b]	
Diameter Surface area	Volume Charging time	Cation	Mean Joule input[c] (mW)	Excess[d,e] (mW)	Mean Joule input[c] (mW)	Excess[d,e] (mW)
6 mm	0.28 cm^3	Li	820	−45 ± 50	960	12 ± 57
3.9 cm^2	870 h	Na	590	14 ± 71	980	57 ± 44
4 mm	0.19 cm^3	Li	800	−19 ± 28	960	−20 ± 29
3.5 cm^2	810 h	Na	610	−55 ± 53	960	−11 ± 49
2 mm	0.094 cm^3	Li	780	40 ± 53	930	61 ± 26
3.1 cm^2	740 h	Na	620	−12 ± 38	940	36 ± 40
1 mm	0.071 cm^3	Li	830	−46 ± 64	980	43 ± 64
2.8 cm^2	670 h	Na	620	−18 ± 60	980	−4 ± 35
Mean excess	$\pm(\sigma/\sqrt{n})$ (mW)[f]			−18 ± 4 (n = 143)		+21 ± 4 (n = 146)

(*b*) IHF and isothermal calorimeters: 0.1M LiOD

	Pd					
Calorimeter type	Type Surface area	Total charging time Volume	Current (mA cm^{-2})	Time (h)	Joule input[c] (W)	Excess[d] (mW)
IHF	2-mm rod[a]	797 h[l]	156	231	2.341	78 ± 77[m]
	3.1 cm^2	0.16 cm^3	156	206	2.380	27 ± 48
			219	187	3.842	−1 ± 58
			279	173	5.188	−55 ± 101
	Cast[g]	797 h[l]	35	236	2.172	100 ± 40
	14 cm^2	0.88 cm^3	36	201	2.219	47 ± 36
			49	187	3.489	−31 ± 51
			62	173	4.863	−120 ± 86
Isothermal	2-mm rod[a]	284 h[b]				
	1.3 cm^2	0.063 cm^3	159	284	0.925[o]	−10 ± 12[n]
	Cast beads[h]	355 h[b]	50	7	0.985	33 ± 12
	5 cm^2	0.5 cm^3	30	15	0.415	11 ± 11
			70	7.8	1.796	5 ± 12
			20	15	0.214	10 ± 11
			100	8.6	3.405	−5 ± 12
			120	270	4.825	2 ± 13
			40	8.1	0.675	7 ± 12
			80	5.7	2.311	9 ± 12
			200	4.8	12.314	−10 ± 12
			160	4.1	8.140	−17 ± 12
			220	4.8	14.675	−9 ± 12
	Melt-spun-ribbon[i]	74 h[l]				
	74 cm^2	0.35 cm^3	14	74	8.991	64 ± 17
	2-mm rod[j]	323 h[l]	152	70	1.091	7 ± 11
	1.3 cm^2	0.066 cm^3	530	253	10.358	15 ± 14
	8-mm bar[k]	520 h[l]				
	28 cm^2	1.5 cm^3	30	520	8.974	36 ± 15

[a] Johnson Matthey (JM) 'Specpure'; drawn from sintered stock prepared from high-purity powder.

[b] D_2O from Harwell reference stock, contains 13 kBq ml^{-1} tritium.

[c] Mean Joule input power supplied to cell (see Fig. 2 legend). Values for IHF and isothermal calorimeters have been corrected for heat loss that is due to evaporation (see Fig. 1 legend).

[d] Excess power = measured cell output power − calculated Joule input power.

[e] (Mean ± 1σ) of values calculated after each refilling to the reference level, excluding first 10,000 minutes of polarization (see text).

[f] Mean and standard deviation of the mean calculated for all H_2O data points and all D_2O data points.

[g] Specially produced material supplied by JM—prepared from cast Pd stock that was argon-arc melted into rod form using a gravity casting process. The rods were subsequently sliced and bent to decrease the loading time required (maximum distance from bulk to surface ~1 mm). The sample was cleaned using acetone, 10% HCl and distilled water.

[h] 'Specpure' Pd arc melted three times under argon on a water-cooled copper hearth.

[i] A variety of ribbons prepared (JM) by melt spinning of cast or sintered Pd. A proportion of the ribbons were heat treated (JM) for 20 min at 100 °C under 10% H_2/N_2. Ribbon thickness, 125 μm.

[j] Type [a] that was subsequently vacuum degassed at 1,200 °C, and loaded with deuterium at a pressure of 40 bar. The sample was cooled to liquid-nitrogen temperature before transferring to the calorimeter to minimize loss of D_2.

[k] Sintered high-purity bar, sliced and bent to decrease the loading time required (maximum distance from surface to bulk ~1 mm).

[l] D_2O from Aldrich Chemical Co., contains ~15 kBq ml^{-1} tritium.

[m] Mean and standard deviation of all data points after temperature stabilization: data point every 3 min.

[n] Standard deviation given by $\sigma = \sqrt{\sigma_b^2 + \sigma_y^2 + \sigma_c^2}$ where σ_b is the standard deviation of the baseline measurement, σ_y that of the power measurement with the cell running and σ_c is the standard error of estimate of the correction line (see Fig. 1 legend). σ_b and σ_y were typically 6 mW, σ_c was 8.8 mW.

[o] Small error in this particular measurement gave rise to the apparent small endotherm.

rod) was ~45 mW and for an H_2O cell (6-mm rod) ~40 mW. These are small compared with the input power. It is certainly clear that no unusually large power pulses occurred. Given the difficulties in operating these calorimeters, very occasional occurrences of small fluctuations cannot be considered as support for a 'fusion' hypothesis.

The other two types of calorimeters used in this study are described in Fig. 1 legend and results are given in Table 1*b*. We explored a range of different preparations of palladium and of current density, up to nearly 600 mA cm^{-2}. With the improved heat-flow calorimeters (IHF), the sign and magnitude of the power excess varied with Joule input power, but was always <5%. Expressed in terms of the volume of the palladium cathode, this sets a limit of 100-500 mW cm^{-3}. The most accurate calorimeter used was the isothermal calorimeter (minimum-detectable power change ~10 mW; minimum-detectable energy in any brief burst ~40 J). We analysed these results at four-hour intervals by averaging both the Joule input power and the measured output power over a period of ~20 min. Inspection of the data collected every four minutes between the regions of analysis showed no obvious signs of any short heat 'bursts' and we found no trend with time of the measured output power under any of the conditions used. Table 1*b* shows that we obtained thermal balance to better than 20 mW (24-240 mW cm^{-3} Pd). We observed slight thermal excesses (30-60 mW) during the initial charging period of the palladium beads, and during runs with high-surface-area cathodes at high current: it can reasonably be assumed that a small amount of recombination (4% at most) was responsible for this effect.

Neutron counting

We investigated the emission of neutrons from a wide range of cells using three different detector systems (Table 2). The large, high-efficiency detector with which most of the neutron measurements were made is an oil-moderated assembly of 56 $^{10}BF_3$ proportional counters configured as 5 concentric rings[11,12]. The total efficiency for 2.45-MeV d-d neutrons is 44%. We built an automatic cell shuttle mechanism to exchange regularly two nominally identical cells, only one of which was powered. This enabled the background (which is due mostly to cosmic rays, there being no anti-coincidence counter arrangements) and any signal to be counted virtually simultaneously. In operation the cells were exchanged every 5 min, and the data from the 5 rings were recorded separately. Data from a typical run are shown in Fig. 3 as differences in the count rate between the powered and unpowered cell. Although in the particular example shown, two spikes can be seen in the count rate differences from the detector as a whole, it is clear that these spikes are due entirely to the misbehaviour of ring 4, and are therefore spurious.

The details of the cells and results of the measurements are given in Table 2. The lowest limits (2σ) on neutron emission derivable from Table 2 for palladium are 1.5×10^{-2} n s^{-1} g^{-1} or 1.5×10^{-2} n s^{-1} cm^{-2}, if the emission were sustained over the whole run. For titanium, the values are 3×10^{-3} n s^{-1} g^{-1} and 4×10^{-3} n s^{-1} cm^{-2}, although the latter limit is too high because it does not apply to the runs using granules having a large and indeterminate surface area. If we assume the emission to be sustained over only a one-hour period at most, then the limits are, for palladium: 7×10^{-2} n s^{-1} g^{-1} or 4×10^{-2} n s^{-1} cm^{-2}, and for titanium: 8×10^{-3} n s^{-1} g^{-1} or 2×10^{-2} n s^{-1} cm^{-2} (much less in the runs with granules). The neutron-emission rate limits are several orders of magnitude below the rates of $\sim10^4$ s^{-1} reported in ref. 1 and about one order of magnitude below the rate of ~0.4 s^{-1} reported in ref. 2. It is significant that the limits in our work were also obtained for cells in which cold fusion could be expected to be enhanced, in particular by using titanium cathodes in the form of a large (40 g) mass of porous granules to increase both the reaction volume and the surface area, and an electrolyte specifically chosen to promote deuterium loading into the cathode[13] (see Fig. 3). As discussed later, we have not expressed the results in terms of the number of deuterium pairs within metal lattices.

Because the net count rates given in Table 2 are differences between much larger background rates, runs were undertaken with a calibrated ^{252}Cf source (0.024 ± 0.003 fissions s^{-1}) emitting 0.09 ± 0.01 n s^{-1} and a physically identical blank. The result was 0.14 ± 0.05 n s^{-1}, in satisfactory agreement. Furthermore, the expected neutron output of the unpowered UPt_3 cell, which is due to spontaneous fission of ^{238}U, was 0.22 s^{-1}. The value measured in comparison with an empty cell was 0.20 ± 0.06 s^{-1}. Our confidence in the results of the shuttle differences seems justified. Measurements on $CeAl_2$ and UPt_3 cells were included in the hope that high effective electron masses corresponding to these metal crystal lattices would mimic in some way the fusion enhancement effect of 'heavy electrons' such as muons in binding the deuterium nuclei closer together. The palladium-ribbon run with the cell power switched on and off every two hours (which was about eight times the characteristic diffusion time in the ribbon) was undertaken to enhance the appearance of non-equilibrium effects, and results (negative) are given in Fig. 4.

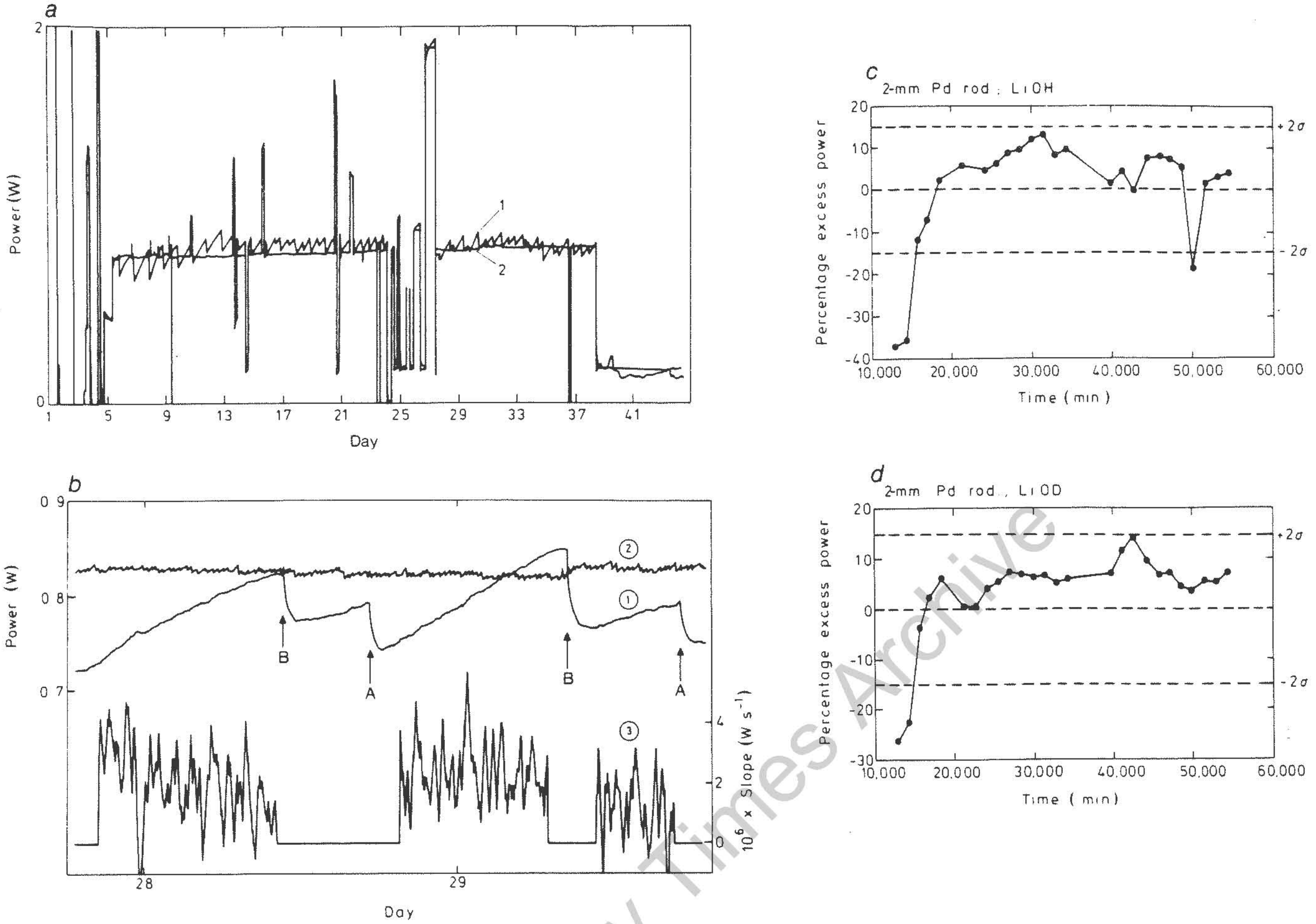
γ-ray counting

As it has been shown that cold proton-deuteron (p-d) fusion is expected to proceed at rates greater by ~8.5 orders of magnitude than d-d fusion[3], we carried out an alternative investigation of electrolytic enhancement of hydrogen-isotope fusion by looking for the $D(p,\gamma)^3He$ 5,488-keV γ-rays from p-d fusion. We used a lead-shielded 113-cm^3 n-type high-purity germanium (HPGe) crystal γ-ray spectrometer to search for any γ-rays in the energy range 0.1-7 MeV emitted by a variety of cold-fusion cells operating with a mixture of light and heavy water. The results, also given in Table 2, are consistent with no γ-ray emission. The lowest limits (2σ) on γ-emission derivable from Table 2 are, for palladium, 7×10^{-3} γ s^{-1} g^{-1} or 1×10^{-3} γ s^{-1} cm^{-2}, and for titanium 1.4×10^{-3} γ s^{-1} g^{-1} or 4×10^{-4} γ s^{-1} cm^{-2}, if we assume that the emission is sustained over the whole run. Jones *et al.*[2] report that the fusion activity may last for only 4-8 h and begin ~1 h after the cell is powered, and in these circumstances typical values for the standard deviation in the γ-ray emission rates are ~0.05 s^{-1} and the 2σ limits for titanium are 4×10^{-3} γ s^{-1} g^{-1} or 3×10^{-3} γ s^{-1} cm^{-2}. Post-run analysis of the hydrogen isotopes taken up by the palladium cathodes with the mixed light and heavy-water electrolytes used gave very satisfactory D:H ratios, lying in the range 1.5-2.5. We note that if the proposed enhancement of the fusion process is as valid for p-d as for d-d fusion then, because of the enhanced tunnelling in the lighter d-p system[3], the γ-ray measurements actually provide a much more stringent limit on the cold fusion process than do the neutron measurements. The interpretation of the results from some of these cells was complicated by the dissolution of gold anodes and the consequent gold deposition onto the cathode: this also would have affected the original work[2].

Tritium enrichment

Fleischmann *et al.*[1] claimed a tritium production rate of $\sim10^4$ atom s^{-1}, commensurate with their reported neutron emission rate. They used a differential technique in which the tritium accumulation in a cell with a palladium cathode was compared with that in a cell with a platinum cathode. They took samples for analysis at regular intervals and maintained a constant total electrolyte volume by the addition of fresh D_2O.

Electrolytic enrichment is widely used to increase the concentration of tritium in water before analysis[14]. Reproducible results require careful control of the electrolysis, as the enrichment factor can vary widely (refs 15, 16 and R. L. Otlet, personal communication): important effects are seen with change of electrode materials, with variation in the condition (activity) of the electrode surface, with the current density (overvoltage) and



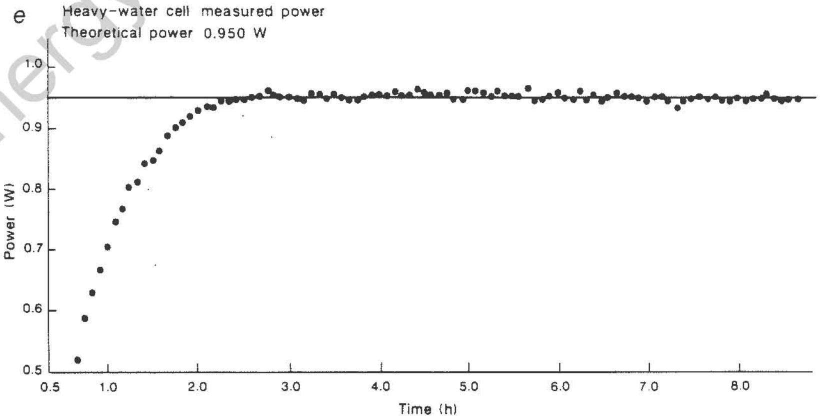


FIG. 2 *a*, Raw data from FPH-type calorimeter containing a 4-mm Pd rod (1.5 cm long, Johnson Matthey 'specpure', drawn from sintered stock) in LiOH elctrolyte. Line 1 represents the output power calculated from the thermistor reading and line 2 represents the Joule input power to the cell, $P_{in} = I(V - V_0)$ where $V_0 = \Delta H_d/2F$ (1.527 V for D_2O and 1.481 V for H_2O, ΔH_d being the enthalpy of dissociation, for example, $D_2O(l) \rightarrow D_2(g) + \frac{1}{2}O_2(g)$). The large step variations are calibrations. *b*, An expanded region of *a*, which emphasizes the sloping baseline. Lines 1 and 2 are as in *a*. Line 3 is the gradient of the apparent output power calculated by differentiation of the data using a seven-point Savitzky-Golay routine[23], stepping one point at a time. At points A the calorimeter was topped up to the reference mark with H_2O pre-warmed to the cell temperature. At points B a volume of liquid estimated from the electrolysis rate was added. *c* and *d*, Results for the *c* and *d*, Results for the two most consistently exothermic FPH calorimeters—0.2 × 3-cm Pd rod in (1) LiOD and (2) LiOH—in the form of percentage excess of apparent output power over the Joule input power. The error bars represent the control limits $\pm 2\sigma$ calculated from the results obtained for all the different cells (see text). *e*, Raw data obtained using the isothermal calorimeter (20 × 2-mm diameter Pd cathode), immediately following the application of the input power, with the line representing the Joule input power and the dots the observed cell power. The response time of this calorimeter is governed by the time to obtain thermal mixing of the cell contents, and is faster than that of the simple calorimeters, despite the large solution volume.

SLOPING BASELINE. The response of the 'simple' FPH calorimeter can be modelled by[10]

$$P_{in} = k\Delta T = (k_{sb} + k_c)\Delta T \qquad (3)$$

where[9],

$$k_{sb} = \sigma A_1[T_i^4 - T_0^4] \approx 4\sigma A_1 T_0^3[1 + 3\Delta T/(2T_0)] = k_{sb,0}(1 + 3\Delta T/(2T_0)) \qquad (4)$$

and[10]

$$k_c \approx \kappa A_2/l = \kappa A_2/(l_0 + IV_m\delta t/(2F\pi r_i^2)) \approx k_{c0}(1 - \alpha\delta t)$$

(P_{in} is the Joule input power, k is the calorimeter constant, k_{sb} and k_c are the contributions to the calorimeter constant of radiative losses through the vacuum jacket and conduction up the glass inner wall respectively, σ is the Stefan-Boltzmann constant, A_1 is the contact area of the solution with the wall of the cell, T_0 is the bath temperature, T_i is the cell temperature, $\Delta T = T_i - T_0$, κ is the thermal conductivity of the glass, l is the distance from the liquid to the point where the inner glass wall comes into contact with the bath and its initial value is l_0, A_2 is the cross-sectional area of the inner glass wall, I is the cell current (300 mA), V_m is the molar volume of cell solution, δt is the elapsed time after refilling the cell, r_i is the internal radius of the glass vessel and $\alpha = IV_m/(2F\pi r_i^2 l_0)$. We measured the calorimeter constant for the cells to be ~0.1 W K^{-1}, with the calculation using equation (4) indicating roughly equal contributions from k_{sb} and k_c. A more complete description of the calorimeter should also take into account the effects of the solution loss on the heat capacity of the calorimeter. It can be shown however that the contribution of this to the sloping baseline is insignificant[10].

with the temperature. Without precautions, the variations in enrichment factor can be more than a factor of two[16]. The claims therefore need to be assessed against this known variable electrolytic enrichment. Surprisingly, Fleischmann *et al.*[1] claimed that there was no electrolytic enrichment in their platinum cell.

If the rates of electrolytic evolution of hydrogen isotopes are written as follows

$$D_2O \rightarrow D_2 + \tfrac{1}{2}O_2 \qquad \text{rate} = r$$

$$DTO \rightarrow DT + \tfrac{1}{2}O_2 \qquad \text{rate} = XSr$$

where r denotes the electrolysis rate, S the tritium-deuterium separation factor and X the mole fraction of tritium, T, in the solution, then the tritium accumulation is given by[10]

$$\frac{X}{X_0} = \frac{1}{(S+\alpha+\gamma)}\left\{1+\alpha+\gamma-(1-S)\exp\left[-\frac{(S+\alpha+\gamma)rt}{N_0}\right]\right\} \tag{1}$$

where the total solution volume is maintained constant by the addition of fresh D_2O (containing a mole fraction X_0 of tritium, directly proportional to the disintegration rate), the ratio of the sampling rate of the solution to the electrolysis rate is α and γ denotes the ratio of the rate of evaporation of the solution to the electrolysis rate—a small correction that may be calculated assuming that the electrolysis gases passing out of the cell are saturated with water vapour. On the timescales of interest, variability in S would give a variable enrichment

$$\delta\left(\frac{X}{X_0}\right) = -\frac{rt}{N_0}\,\delta S \tag{2}$$

If the solution becomes contaminated by hydrogen absorption from the atmosphere, then if S_{obs} denotes the apparent DT enrichment factor derived from the application of equation (1), $S_{obs} = S/[0.5(1+f)]$, where $f = (1-X_H)/(1+X_H(S_{HD}-1))$ with X_H denoting the mole fraction of hydrogen and S_{HD} the HD separation factor (relative rate of reaction of HDO and D_2O). Therefore, as well as the inherent variability from one electrode to another, it is evident that any variability in the amount of hydrogen pickup will give a variation in the apparent enrichment factor:

$$\frac{dS_{obs}}{dX_H} = \frac{S_{HD}}{2[1+X_H(S_{HD}-1)^2]}$$

We found that, unless exceptional precautions were taken (and we believe that we used experimental procedures very similar to those used in ref. 1 (M. Fleischmann, personal communication)), values of $X_H \approx 0.07$ were common. Under these conditions, a change of X_H of only 0.01 would give a change in S_{obs} of 0.02, which could be significant given the smallness of the claimed effect.

Any assessment of whether differential enrichment can be considered to account for the results in ref. 1 depends critically on the value of X_0, which was not reported. Using values[1] of r (1.24×10^{18} atom s^{-1}) and N_0 (14.6×10^{23} atom) we calculate from equation (2) that if X_0 were at the extreme low end of the range for commercial D_2O (3 Bq ml^{-1}), a value of $\delta S = 0.46$ would be required; if it were moderate (10-15 Bq ml^{-1}) a value $\delta S \approx 0.1$ would be needed; if it were high (80 Bq ml^{-1}) $\delta S \approx 0.02$ would suffice.

The applicability of equation (1) was confirmed experimentally, on both platinum and palladium cathodes, in conjunction with calorimetric and neutron-counting experiments (see Fig. 5 legend) using D_2O with an initial tritium content of 13 kBq ml^{-1} (efficiency corrected). The fit of this equation to all the experimental data (Fig. 5) gave, for palladium, $S_{obs} = 0.59$, and for platinum, $S_{obs} = 0.61$. Correction for the uptake of hydrogen, using $S_{HD} = 6$ (ref. 16), gave $S \approx 0.48$, in agreement with previous work and theoretical expectations ($S = 0.46 \pm 0.02$, (refs 14, 15 and D. S. Rawson and R. L. Otlet, personal communication)). For individual electrodes, S_{obs} for palladium varied from 0.46 to 0.65 (corrected S from 0.42 to 0.58), whereas S_{obs} for platinum varied from 0.56 to 0.85 (corrected S from 0.43 to 0.69)—a total range for individual electrodes of $\delta S_{obs} = 0.4$, which is enough to account for the results of Fleischmann *et al.*[1].

It is clear from these results and discussion that more evidence needs to be presented before the tritium accumulation reported[1] can be considered as experimentally reliable evidence for the occurrence of a fusion process.

Materials characterization

With 125-μm palladium foils, hydrogen loadings (determined[10] by hot-extraction mass spectrometry of specimens frozen in liquid nitrogen, and independently by electrochemical extraction) of $H/Pd = 0.95 \pm 0.05$ were achieved in ~1 h at 100 mA cm^{-2} in 0.1 M LiOH solution with a platinum anode. The limit of deuterium loading in 0.1 M LiOD was lower (0.84 ± 0.03). Rods (1- and 2-mm diameter) polarized for extended periods in neutron counting and calorimetry experiments showed $D/Pd = 0.76 \pm 0.06$. It is well known[17] that the equilibrium pressure for a given deuterium loading is higher than that for the same hydrogen loading. Current-interruption methods confirmed that overpotentials in the range 0.8-1 V (ref. 1) were being obtained. From an initial composition >99.9% D_2O, the solutions degraded to ~98·5% in 24 h and to 88-98% (analysis by infrared spectrometry) following electrolysis for many weeks. The resulting ratio H/D in the palladium was 0.02-0.04.

During electrolysis, all of the palladium cathodes became covered with a layer that varied in appearance from a dull tarnish to a dense jet black. In the latter case, loose black material was also formed, which in extreme cases came off during electrolysis, resulting in quite heavy erosion of the cathode. The layer itself evidently represented a modification of the morphology of the cathode at the surface. The formation of a thick black layer was enhanced at high current density, at high temperature, on smaller diameter wire and by frequent abrupt alterations of the current density repeated over a long period. The layer was more noticeable on the outside of a spiral-wound cathode than on the inside. The layer was also more noticeable on cathodes polarized in D_2O than on those polarized in H_2O (perhaps this is related to the greater equilibrium gas pressure for equivalent composition in the Pd-D system), and was different in appearance on materials from different sources. These observations may be explained by the old idea[17] that microfissures, or rifts, develop in palladium to release the mechanical strains resulting from the heavy loading of hydrogen, together with the assumption that any such effect would depend on the stress state of the metal surface and its microstructure.

Lithium was present in the surface layer on cathodes used in LiOD, and analysis by secondary-ion mass spectrometry (SIMS) apparently showed a concentration profile extending about 1 μm into the metal. SIMS images showed that the lithium was not uniformly distributed, however, and it seemed likely that it was trapped in microfissures in the surface layer.

Surface analysis showed a number of other species on and in the surface layer, notably small quantities of platinum and traces of copper, zinc, iron, lead and silicon: platinum would have originated from the anode and silicon from the glass container. No doubt the majority of the other contamination would have come from the solution: the levels found (a few atom per cent, confined to the surface layers) were consistent with deposition by extended electrolysis from a solution of concentration around 10^{-9}–10^{-10}M. One possible criticism is that low levels of such deposition could poison any essential catalytic activity: we therefore used pre-electrolysis in several experiments, in an attempt to lower the surface contamination. We either made repeat experiments on the same solution, simply changing the cathode, or treated the solution beforehand in a separate cell.

Because of claims that an unusual mechanism might lead to

TABLE 2 Measured neutron and γ-ray emission rates for cold fusion electrolytic cells

(*a*) Neutron emission rates

	Cathode				Details of electrolysis			
Material	Mass (g)	Surface area (cm^2)	Electrolyte	Anode	Current (mA)	Typical voltage	Duration (h)	Measured neutron yield[w] (n sec^{-1})
Pd rod 2 mm[a]	3.4	5.7	0.1M LiOD	Pt wire	360	5	914	These cells monitored initially by low-efficiency n-detectors with lower detection limit of ~100 s^{-1}. Later moved to a dual-cavity neutron detector[10] with lower detection limit ~2 s^{-1}. No detected n-emission.
Pd wire 1 mm[a]	0.94	3.1	0.1M LiOD	Pt wire	200[r]	4	916	
Pd wire 1 mm[b]	0.94	3.1	0.1M LiOD	Pt wire	200	4	916	
Pd wire 1 mm[b]	1.4	4.7	0.1M LiOD	Pt wire	300[s]	5	917	
Pd wire 1 mm[c]	0.47	1.6	0.1M LiOD	Pt wire	750	11	856	
Pd plate 1 mm[d]	7.5	13.5	1M LiOD	Pt sheet	2,000	10	307	
Pd foil	0.5	8.0	0.1M LiOD	Vitreous C	400-120[t]	4-20	142	
Pd/22Ag[e]	1.6	3.8	0.1M LiOD	Pt wire	380	5	859	
Pd/22Ag[e]	1.6	3.8	0.1M LiOD	Pt wire	380[u]	5	547	
Pd foil[f]	0.075	1.0	0.1M LiOD	Pt wire	30	3	87	−0.019 ± 0.042
Pd foil[g]	0.075	1.0	0.1M LiOD	Pt wire	250	7	6	0.00 ± 0.08
Pd foil[h]	0.075	1.0	0.1M LiOD +17 mM Na_2S	Pt wire	250	7	16	0.04 ± 0.11
Pd foil[i]	0.075	1.0	0.1M LiOD	Pt wire	250	8	16	0.005 ± 0.063
Pd foil[j]	0.075	1.0	0.1M LiOD	Pt wire	250	8	16	0.051 ± 0.061
Pd foil[k]	0.075	1.0	0.1M LiOD	Pt wire	1,000	15	56	0.068 ± 0.061
Pd foil	0.63	8.0	0.1M LiOD	Pt foil	1,000	14	17	−0.110 ± 0.082
Pd foil	0.63	8.0	0.1M LiOD	Pd foil	1,000	18	22	0.012 ± 0.079
Pd ribbon[l]	0.45	0.92	0.1M LiOD	Pd foil	600[v]	15	110	−0.063 ± 0.096
Pd foil	0.60	8.0	0.1M LiOD	Au wire	1,000	12	43	0.068 ± 0.058
Pd pellet[m]	3.0	2	0.1M LiOD	Pt foil	650	18	68	0.007 ± 0.042
Pd pieces[n]	4.4		0.1M LiOD	Pd foil	500	8	88	0.012 ± 0.033
Pd pieces	4.4		0.1M LiOD	Pd foil	500	15	88	0.003 ± 0.046[x]
Ti foil	0.038	1.0	0.1M D_2SO_4	Au wire	250	5	34	−0.091 ± 0.054
Ti foil	0.038	1.0	0.1M D_2SO_4	Au foil	1,000	5	3	−0.22 ± 0.12[y]
(continued)			0.1M D_2SO_4 +0.02M $PdCl_2$		1,000	5	4	−0.22 ± 0.12[y]
Ti rod	2.2	4.3	Jones[q]	Au foil	100	4	3	−0.06 ± 0.15[y]
(continued)					500	6	2	−0.42 ± 0.21[y]
Ti granules[o]	0.5		0.1M D_2SO_4	Pt wire	250	7	22	0.00 ± 0.072
Ti granules	0.5		0.1M D_2SO_4 +3 mM $Na_4P_2O_7$	Pt wire	250	7	66	0.044 ± 0.054
Ti granules	40		0.1M D_2SO_4	Pt wire	250	25	22	−0.009 ± 0.058
Ti granules	40		0.1M D_2SO_4 +3 mM $Na_4P_2O_7$	Pt foil	250	25	22	−0.010 ± 0.060
Ti granules	17		Jones	Au foil	500	26	24	0.021 ± 0.054
Ti granules								
Ti/6Al/4V	2.8	15.0	0.1M D_2SO_4	Pt wire	670	5	87	−0.003 ± 0.031[x]
Ti/6Al/4V[p]	2.7	14.3	0.1M D_2SO_4	Pt wire	660	5	19	0.031 ± 0.044[x]
TiFe granules	1		0.1M D_2SO_4	Pt foil	250	8	24	0.12 ± 0.06
$CeAl_2$ granules	11		0.1M LiOD	Pt foil	500	22	22	−0.051 ± 0.056
UPt_3 granules	57		0.1M LiOD	Pt foil	500	20	94	0.10 ± 0.06

(*b*) γ-ray emission rates

	Cathode				Details of electrolysis			
Material	Mass (g)	Surface area (cm^2)	Electrolyte[z]	Anode	Current (mA)	Typical voltage	Duration (h)	Measured 5,488-keV γ-ray yield[aa] (γ s^{-1})
Pd foil	0.58	7.5	0.1M LiOH	Pt foil	450	9	45	$(-4.9 \pm 7.43) \times 10^{-3}$
Pd foil	0.74	9.5	0.1M LiOH	Pt foil	510	9	164	−0.011 ± 0.004
Pd sheet	7.7	12.9	0.1M LiOH*	Pt foil	200	4.6	50	−0.017 ± 0.012
Pd rod	3.1	2.3	0.1M LiOH	Au foil	1,000	26	17	0.008 ± 0.011
Ti foil	0.089	2.0	Jones*	Au foil	320	5	42	−0.022 ± 0.011
Ti foil	2.2	32	Jones	Au foil	1,000	4	12	−0.015 ± 0.034
Ti foil	2.2	32	Jones	Au foil	1,000	4	6	0.012 ± 0.007
Ti granules	25		Jones	Au foil	710	13	8	0.032 ± 0.036
Ti granules	25		0.1M H_2SO_4	Au foil	400	20	25	−0.019 ± 0.018
Ti granules	25		0.1M H_2SO_4	Au foil	765	16	16	0.017 ± 0.022
UPt_3 granules	42		0.1M LiOH	Pt foil	300	31	166	$(3.1 \pm 6.0) \times 10^{-3}$

[a] Cells provided by M. Fleischmann. Cathodes analysed for H, D after use—results H/D: 0.01, 0.02 D/Pd: 0.84, 0.72.

[b] Cathode wound into a tight spiral. Examined initially in the large n-detector, without shuttle, for 147 h with estimated detection limit 1 n s^{-1} and a further 5 h with detection limit 0.2 n s^{-1}. Surface of cathode then rubbed with S before further use.

[c] Cathode cleaned with emery paper after 300 h.

[d] Cathode examined initially in the large n-detector, without shuttle, in 0.1M LiOD for 183 h; estimated detection limit 1 n s^{-1}. Abraded with 400-grit SiC paper before use.

[e] Tubes from D storage system for Tokamak.

[f] Cathode analysed for H, D after use—results: H/Pd: 0.03 D/Pd: 0.80.

[g] Electrolyte of the above run reused.

[h] Cathode and electrolyte of the above run reused; Na_2S added as concentrated aqueous solution.

Foil vacuum degassed 1,000 °C before use. Analysed for H, D after use—results: H/Pd: 0.02 D/Pd: 0.83.

[j] Foil dipped in Na_2S (concentrated solution) before use. Analysed for H, D after use—results: H/Pd: 0.05 D/Pd: 0.80.

[k] Cathode cut in half and analysed for H, D after use—results: H/Pd: 0.02, 0.02 D/Pd: 0.85, 0.78.

[l] Melt-spun ribbon provided by Johnson-Matthey Technology Centre.

[m] Arc remelted twice; electrolytically charged for 1 month in 0.1M LiOD then frozen in liquid nitrogen, dipped in concentrated Na_2S solution and transferred to n-counting cell.

[n] Four 1-2-mm-thick discs of different types of Pd (Johnson-Matthey) spot welded to Pd wires plus strained Pd Wire.

[o] Electrolyte reused after a previous run with a Ti cathode and Pt anode.

[p] Material heated to 900 °C then quenched in water before use.

[q] AuCN in Jones electrolyte replaced with $NaAuCl_4$

[r] Current on for 36 h then changed between 200 mA and 20 mA every hour.

[s] Current on for 36 h then changed between 300 mA and 30 mA every 5 min.

[t] Current changed slowly over range shown during electrolysis period as anode disintegrated.

[u] Current on for 36 h then cycled off for 10 h and on for 2 h for period of 270 h. Then 5,000 s at 380 mA cathodic and 4,000 s at 10 mA anodic for rest of period.

[v] Current cycled on/off every 2 h. Neutron yield is for 'on' cycle.

[w] The errors assigned (1σ, calculated over full run duration) vary somewhat because in an attempt (mostly in the earlier stages of the programme) to cover as wide a range of cell configurations as possible, pairs of unpowered + unpowered and powered + unpowered runs were not always carried out, and consequently allowance has to made for slight differences in overall cosmic-ray neutron detection efficiencies caused by slight differences between the two nominally identical cells.

[x] These data were obtained using a different data acquisition system to drive the shuttle and accumulate data, set up to look for neutron bursts[10].

[y] Cells exchanged every 5 min by hand.

[z] All electrolytes used for γ-ray work were 50:50 $H_2O:D_2O$ except * which were 41:59 $H_2O:D_2O$.

[aa] For the present measurements, the system was calibrated with a set of standard γ-ray sources and a $^{238}Pu/^{13}C$ (α, nγ) source emitting 6,129-keV γ-rays. The cells were positioned such that their cathodes were as close as possible to the detector crystal, and the detection efficiencies were calculated by integrating previously measured[21] point efficiency functions over the cathode volume. Peaks were searched for at 5,488, 4,977 and 4,466 keV corresponding to the expected location of the full energy, single-escape and double-escape peaks using the method recommended in ref. 22.

nuclear reactions proceeding predominantly non-radiatively to ^{4}He, four cathodes (Table 2, first four entries) were analysed by vacuum fusion/mass spectrometry. The high vapour pressure of palladium at the melting point caused difficulties, so internal standards were prepared by ion implantation of 10^{13} and 10^{15} atoms of ^{4}He into samples cut from the cathodes. Detection limits for ^{3}He and ^{4}He determined in this way were $\sim 8 \times 10^{10}$ atoms per sample—$(1\text{-}10) \times 10^{11}$ atom g^{-1}. We found no ^{3}He or ^{4}He. The expected level, if fusion had been occurring at the rate reported in ref. 1, was $\sim 10^{16}$ atom g^{-1}.

Evolution of hydrogen at a titanium cathode resulted, as is well known[13], in a dense network of hydride precipitates, observable by standard metallographic methods, penetrating below the surface. In 0.1M D_2SO_4 electrolyte at 100 mA cm^{-2}, the network penetrated ~30 μm in 1 h. Precious-metal deposition inhibited the electrolytic uptake, presumably by lowering the overvoltage and promoting gas evolution. Electrolysis in the 'brew' used by Jones *et al.*[2] resulted in hydride precipitates confined to the grain boundaries; other experiments showed that the presence of $PdCl_2$ in the electrolyte caused this effect, presumably as a consequence of palladium plating on the cathode. It is clear that expressions of the fusion rate that assume that the cathode has composition TiD_2 are completely misleading: metallography shows that the number of deuterium pairs is far fewer, hence the claimed fusion rate per deuterium pair is far higher than the figure given, and so the results are even more difficult to reconcile with expectations than had been implied[2].

Discussion

The interest in cold fusion has generated a large number of neutron counting experiments. It is well known that, in general, it is inadvisable to measure the signal + background and background of counting measurements at different times and in different physical locations (as in ref. 1), because of unexpected systematic variations. This is especially true for low-count-rate experiments. Compensating for variation of the background rate and assessing appropriate errors for the procedure chosen are particular problems. The work of Jones *et al.*[2] can be criticized for such errors[18]. It is notable that in ref. 2, only one run in fourteen showed a significant effect and then only because this particular run was assigned a smaller counting error than the others. Here we have attempted to minimize, by the shuttle procedure, uncertainty about background and counter variability and about error calculation. Given some of the more spectacular claims that have been made, we note that further caution is advisable because of the notorious sensitivity of $^{10}BF_3$ and ^{3}He proportional counters to humidity and of counter-amplifier systems to earth loops. In our work the neutron detectors were segmented, and the relationship between signals from the segments was well known, so spurious effects giving inconsistent signals from the segments could be identified.

Failure to reproduce the effects has been attributed by some to the need for rigorous exclusion of hydrogen and claims have been made that palladium electrodes must be cast and carefully degassed before use, to remove all traces of carbon and hydrogen impurity which might decorate dislocations or other high-energy

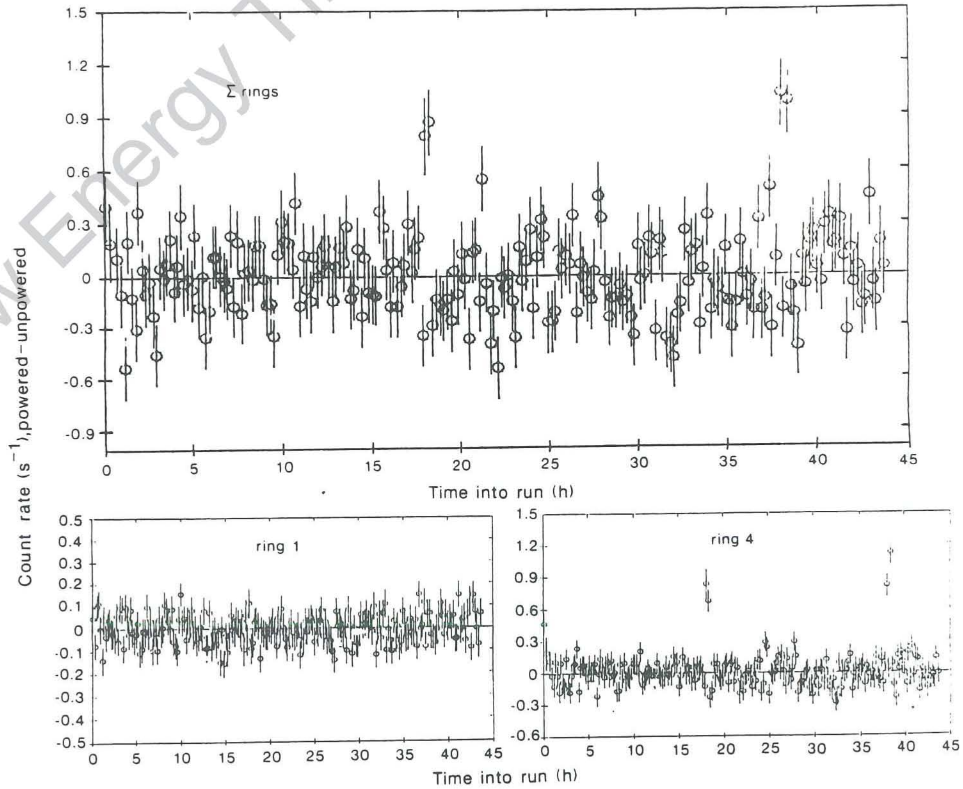


FIG. 3 Data from a typical run on the large high-efficiency neutron detector. These show the differences between count rates for powered and unpowered cells resulting from successive alternate shuttle positions for 40 g of titanium granules in 0.1M D_2SO_4. The errors shown are 1σ errors for each five-minute counting period. In addition to results from the detector as a whole, results from two of the five rings of 2-inch diameter 107-cm active length $^{10}BF_3$ proportional counters are also shown. The counts in rings 2, 3 and 5 (not shown) were very similar to those in ring 1. The apparent bursts were seen only in ring 4 and are therefore a spurious effect. A tube (90-mm inner diameter) passes through the centre of the detector, and the shielding consists of 6 inches of borated resin, 1 mm of cadmium and 2 inches of lead. The neutron detection efficiency is high and is largely independent of neutron energy, varying from 48% for 0.5-MeV (Am/Li) neutrons to 40% for 4.2-MeV (Am/Be) neutrons. The background count rate is 4-5 count s^{-1} mostly neutrons from cosmic rays. Each of the five rings of counters has its own independent pre-amplifier, pulse-shaping amplifier and discriminator, and the mean energy of neutrons counted can be obtained from the ratio of counts in the outermost ring of counters (ring 5) to counts in the innermost (ring 1). Because neutron counts are distributed over five rings, and because of the 135-μs-mean time to capture, neutrons emitted simultaneously from a source as a burst are counted separately (as seen with the ^{252}Cf source, for example). The pre-amplifiers, high voltage components and insulators are all contained within a desiccated electrically screened box, and we eliminate earth loops and induced electromagnetic pick-up ('aerial') effects from external coaxial-cable runs to the PDP-11/45 data-acquisition computer by using isolating high-frequency pulse transformers and by winding the coaxial cables many times around ferrite rings. The neutron detector and its immediately associated electronics are located in a temperature-controlled air-conditioned blockhouse with two-foot-thick concrete walls and roof.

sites (ref. 19 and R. A. Huggins, Workshop on Cold Fusion, Santa Fe, May 1989): this is however inconsistent with the postulate of a 'fusion' origin for the effect because, at low energies, p-d fusion is expected to be significantly faster than d-d fusion[3]. Furthermore, the original reports[1,2] did not mention any special precautions to exclude atmospheric water vapour apart from careful covering of the electrolytic cells. We followed similar procedures, and found that degradation of the heavy water by exchange with atmospheric moisture occurred quite rapidly. Alternatively, it is claimed that it is essential to maintain a high current density for a considerable period (A. J. Appleby, Workshop on Cold Fusion, Santa Fe, May 1989) although it is not completely clear whether these latter claims are in fact reproductions of the effect reported in ref. 1 or are something different. In our neutron counting experiments, fresh dislocations were introduced by plastic deformation, some counting experiments were carried out at current density as high as $1\ \mathrm{A\ cm^{-2}}$ (Table 2) and in one experiment in the isothermal calorimeter a high current density was maintained for a considerable period (Table 1). Trace deposition of platinum on the cathode, supposedly causing a lowering of the overpotential for deuterium evolution and hence a lowering of the attainable deuterium level in the cathode, has also been suggested as an explanation for irreproducibility (M. Fleischmann, personal communication). We observed no effect when we used palladium anodes in our neutron counting experiments.

Timescales for hydrogen and deuterium loading consistent with the expected diffusion time (x^2/D where the diffusion coefficient, $D = 10^{-7}\ \mathrm{cm^2\ s^{-1}}$ and x is the radius or half-thickness of the specimen) were measured[10] and nuclear counting and calorimetric experiments were always conducted over periods much longer than this. Furthermore, in the process of electrolytic loading of palladium, there will clearly be a concentration gradient, a moving phase boundary and the outer atomic layers of the metal will be saturated (possibly supersaturated) with deuterium[17]. It might be expected, therefore, that sufficiently sensitive equipment would detect any fusion process well before the material is completely loaded. Because our neutron detection sensitivity was $\sim 10^5$-10^6 times greater than that of Fleischmann *et al.*[1], it seems unlikely that any greatly enhanced fusion process associated with the absorption of deuterium into palladium, giving rise to neutron emission, is occurring. We are, of course, aware that it is always possible to construct essentially untestable theories involving hypothetical special conditions of the metal or of its surface. Careful characterization of materials for which positive results are claimed is therefore of great importance.

It has been argued that the neutron branch of the d-d reaction might be completely suppressed in favour of the (t+p) branch and it has been further argued (S. Pons, personal communication) that the tritium produced as a result of a nuclear process inside the electrode need not necessarily exchange with the electrolyte and might not therefore be detected. However, the other product of such a nuclear process, a high-energy proton, should be detectable by its interaction with the lattice, including neutron emission: we estimate, knowing the rate of energy loss of the protons and by comparison with (p, n) reaction cross-sections for neighbouring elements, a yield of $\sim 10^{-6}$ neutron per proton, implying a neutron yield in our counting experiments of as much as $10^4\ \mathrm{s^{-1}}$ if fusion at the rate reported in ref. 1 were to proceed entirely through the (t+p) branch.

In view of the rather large d-d separations in both PdD and $\mathrm{TiD_2}$, it might be argued that fusion requires some non-equilibrium state in the lattice—perhaps at the α/β phase boundary in palladium or at the tips of the growing $\mathrm{TiD_2}$ needles or at a lattice defect—where the d-d or p-d separation could be greatly reduced. Here we created non-equilibrium situations by pulsing the current but we detected no neutron emission (Fig. 4).

Some explanations of the apparent excess heat production have emphasized recombination processes at catalytic metal surfaces[20]: apart from occasional explosions, however, which did not result in any detectable neutron emission, we found, by comparison of the volume of water added to maintain the cell volume with the electrolysis charge passed, that this was not a significant effect (in agreement with others[9]) Recombination in the gas space above the liquid, either on exposed cathode surface or catalysed by colloidal metal particles eroded from the cathode, might however account for some of the observations of bursts of heat recently reported (M. Fleischmann and S. Pons, Electrochemical Society Meeting, Los Angeles, May 1989). In discussing the claims in ref. 1, we prefer to focus on characteristics of the 'simple' Fleischman, Pons and Hawkins (FPH) calorimeters, because we have only observed small effects (at the level of the inherent uncertainties), which might mistakenly be claimed as arising from cold fusion, in the one type of calorimeter (FPH type) that has major calibration difficulties. Cells using the same electrode and electrolyte materials operated in calorimeters that did not have these problems exhibited none of these effects.

There are two points regarding the calibration that could have a profound effect on the apparent results obtained with FPH-type calorimeters: the first concerns when the calibration is performed and the second how it is performed. Concerning the first point, it seems from our work and that of Lewis *et al.*[8] that a calibration performed during the first 10,000 min of electrolysis could be seriously in error and lead to an erroneous conclusion that subsequently, rather than being in balance, the cells were exothermic. Concerning the second point, Fleischmann *et al.*[1] describe calibration using the internal-resistance heater, by measurement of Newton's-law-of-cooling losses. Typically, this procedure might involve the application of power to the heater while electrolysis was occurring, following the temperature-time trace until a steady state was obtained, then switching the heater off and following the cooling curve. This procedure gives an approximation to the differential calorimeter constant, $k_d = d(\Delta P)/d(\Delta T)$ (where P is power and T is temperature) at the operating temperature of the cell and can give rise to errors in two ways. First, because any calibration sequence would require

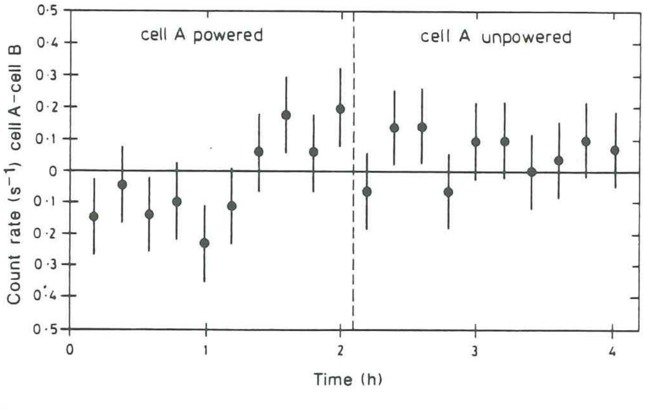


FIG. 4 Time dependence of neutron emission from an electrolytic cell with melt-spun Pd ribbon cathode and Pd foil anode, with power alternately turned on for deuterium loading and off for relaxation every 2 h by the data-acquisition computer: see Table 2 footnote *l*. The total length of the run was 110 h, and the sums of the 'on' and 'off' data are shown. After 90 h of running, we added $1\ \mu\mathrm{M\ Pb^{2+}}$ (to poison the surface by Pb deposition) to the solution. There was no difference between the data in the presence and absence of $\mathrm{Pb^{2+}}$, and so data for the whole run was combined. This run was undertaken to enhance the appearance of the non-equilibrium effects discussed in the text. It is assumed that the ribbon would have had a high density of grain boundaries, dislocations and other lattice defects. Because the 'on' and 'off' periods were several times the characteristic diffusion time for the ribbon, it is assumed that the composition was cycling in the β-phase region between the fully loaded condition ($\mathrm{D/Pd} \approx 0.83$) and the limit of the ($\alpha+\beta$) phase field ($\mathrm{D/Pd} \approx 0.65$).

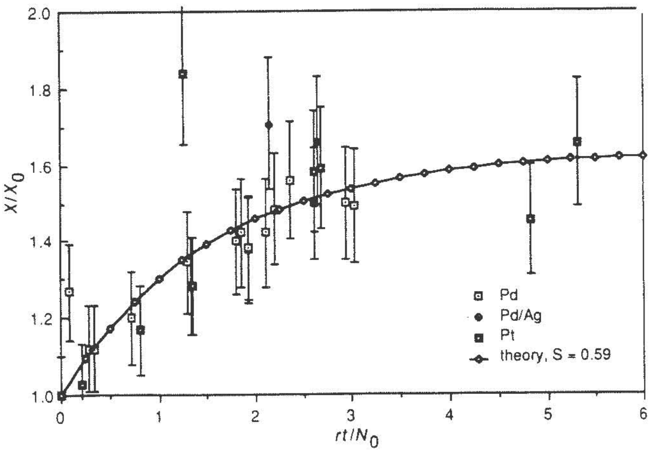


FIG. 5 Tritium enrichment by electrolysis in open cells at constant volume, with both Pd and Pt cathodes: relative count rate X/X_0 against amount of electrolysis rt/N_0 (symbols defined in the text). Initial count rate (efficiency corrected) was $13.0 \pm 0.6\,\text{kBq ml}^{-1}$. Errors are 1σ. The Pd cells were the FPH calorimeters, sampled after 490–660 h, the cells in the first section of Table 2 (footnotes *a–e*) sampled at the end of the run and one cell from the high-efficiency neutron detector—footnote *k* in Table 2. The line is the fit to equation (1).

5–10 h, extrapolation would be required to obtain the correct value at the reference liquid level: an estimated error of 20% or more could result. Furthermore, because the evaporation of the cell contents increases markedly with increasing temperature, the baseline slope would increase with increasing temperature and the effects of this sort of error would become correspondingly more marked: the claimed effects were indeed greatest in the cells run at the highest input power. Second, these calorimeters are nonlinear (equation (3), Fig. 2 legend), with the effect being significant when the temperature gradient is large: they cannot be described by a simple Newton's-law-of-cooling constant. If a differential calorimeter constant is used to derive the input power, the calculated output would be (from Fig. 2 legend, equation (3))

$$P_{\text{app}} = k_{\text{d}}\Delta T = (k_{\text{sb},0} + k_{\text{c}})\Delta T + 3(k_{\text{sb},0}/T_0)(\Delta T)^2$$

so that, in comparison with the correct output (equation (3))

$$P_{\text{app}} - P = 3k_{\text{sb},0}(\Delta T)^2/2T_0)$$

If the power applied to the heater is significant compared with the electrolysis power then this error will be even greater. Back calculation from the data given in ref. 1 shows that the claimed effects were largest for Joule input powers on the order of 6 W. For our FPH-type calorimeters, this would have given $\Delta T \approx$ 50–60 °C. Therefore, had these cells been calibrated in this way, an apparent heat excess on the order of 0.8–1 W would have been observed. Our method of calibration (see Fig. 1 caption) considerably reduced the effect of these sources of error. The original report[1] was clearly preliminary in nature, and it is evident from the above that the claims made therein cannot be assessed in the absence of a detailed description of the experimental procedure used and of the methods used to compensate for the systematic errors inherent in the use of a simple calorimeter.

We feel that our work has served to establish clear bounds for the non-observance of cold fusion in electrolysis cells, under carefully controlled and well understood experimental conditions and using well characterized materials. Further details are given in ref. 10. Claims of observations of cold fusion ought now to meet similar standards of data analysis and materials characterization so that a proper assessment can be made. □

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To: RLG2@YKTVMV.BITNET
X-VMS-To: in%"RLG2@YKTVMV.BITNET"

State University of New York at Stony Brook
Stony Brook, NY 11794-3400

Jacob Bigeleisen
Professor
Chemistry
516-632-7905
10-Dec-1989 02:52pm EST

FROM: JBIGELEISEN
TO: Remote Addressee (_RLG2@YKTVMV.BITNET)

SUBJECT: Tritium at Los Alamos

Dear Dick,

I concur with your evaluation of Storms work at Los Alamos on "finding" tritium. God knows there is plenty of tritium around at Los Alamos and it is hard not to find it. I made this quite clear in my letter of 10 November to Storms. I trust you have now received a copy of that letter from Bill Woodard.

Unfortunately, poor work on cold fusion continues. I now have a manuscript of Bockris' to review. Some time ago he proposed an explanation for the lack of neutrons as a result of the fact that the high electric field at his dendrites interact with the dipole moment of the deuteron and by an Oppenheimer Phillips process give only the T + H branch. I told him the deuteron has no dipole moment. He has now discovered the quadrupole moment of the deuteron and his electric field operates on the quadrupole moment! He is hopeless. His work is upported by EPRI and the Welch Foundation. I am thinking about sending them a bill for consulting on Bockris' work.

Best wishes to you and Lois for the holiday season and the NEW YEAR.

JAKE

Received: from pupgg.princeton.edu by IBM.COM (IBM VM SMTP R1.2.1MX) with TCP; Sat, 09 Dec 89 15:41:57 PST
Date: 9 Dec 89 18:41:00 EST
From: happer@pupgg.princeton.edu
Subject: Attempt by WH to establish computer mail link with RLG.
To: "rlg2" <rlg2@ibm.com>

Dear Dick,
Thank you for the letter from Robert Bass. Wow! I am trying to join the modern world by establishing a computer mail address. Please let me know if this gets through to you.

Regards, Will Happer

COMPTE RENDU DE MISSION AUX ETATS UNIS SUR LA FUSION FROIDE. (Juillet 1989)

J.P.BABUEL-PEYRISSAC,(C.E.A.,IRDI,DESICP,Saclay)

E.ROTH,(Professeur au CNAM,chargé de mission au C.E.A.,DESI

J.BOUCHEZ,(C.E.A.,IRF,DPhPE,Saclay)

C.A.PHILIS,(C.E.A.,DAM,PTN,Bruyères le Châtel)

J.L.TRUFFIER,(C.E.A.,DAM,DETN,Bruyères le Châtel)

PLAN

--Introduction.

--Visite des laboratoires.

--Renseignements sur laboratoires non visités.

--Discussion des travaux.

--Conclusions.

--Liste des documents rapportés.

--Annexe.

INTRODUCTION

Cette mission a été faite à la suite d'une demande des Communautés Européennes, pour réaliser une enquête sur les travaux concernant ce qui est actuellement appelé la "Fusion froide".

Cette enquête visait plus spécialement les laboratoires américains indiquant des résultats positifs, le cas de Fleischmann et Pons étant mis à part.

Nous avons successivement visité:

- Les 10 et 11 Juillet, les divers laboratoires de Texas A & M University, College Station, Texas, en particulier ceux des Professeurs Bockris, Appleby, Martin, Wolf, Marsh.
- Les 12 et 13 Juillet, les laboratoires de Los Alamos, en particulier ceux des Dr. Menlove et Storms.
- Le 14 Juillet, le laboratoire du Professeur Huggins à Stanford.
- Le 15 Juillet, nous avons rendu visite au Dr. Garwin à San Diego. Le Dr. Garwin a été membre du panel très récent organisé par le D.O.E. à Washington sur la Fusion froide. Ceci nous a permis d'avoir une opinion plus nuancée sur les résultats qui nous avaient été précédemment présentés.

VISITE DU LABORATOIRE DU PROFESSEUR BOCKRIS.(Texas A & M University)

Nous avons rencontré:

R. Kainthla,
N.J.C. Packham,
L.Kaba,
F. Cheng

Le professeur Bockris était en visite à Madrid où des résultats intéressants semblent avoir été obtenus.

Le laboratoire réalise des expériences d'électrolyse. Les principaux travaux effectués sont:

-Préparation des électrodes en palladium.
Selon le cas,le palladium est utilisé tel quel,ou bien attaqué par acide,ou bien recuit à 800° sous vide de dix moins six torrs de 12 à 24 heures.On mesure sur certains échantillons le spectre Auger avant et après recuit.Il montre,avant,la présence de pics de C,S,O et, après,celle de Si associée à la diminution des précédents. Le silicium apparait par ségrégation du volume vers la surface.

-Préélectrolyse et chargement.
Le chargement en deutérium de la cathode se fait par une électrolyse dans une cellule à anode de nickel,à une intensité de 50 à 60 mA/cm2.Elle est très longue et peut durer de une semaine à un mois.Une vingtaine d'électrodes sont préparées simultanément.Bockris (doc. n° 5)insiste beaucoup sur cette étape,longue mais indispensable pour réussir.Il semble bien que ce soit là la méthode employée par Pons et Fleischmann qui sont d'ailleurs spécialistes de cette façon d'opérer.Par pesée,on détermine un rapport D/Pd de 0,8 au moins.Dans une lettre de protestation au directeur de la revue "NATURE",(Doc. n° 4),Bockris insiste sur le fait que ces durées de chargement étant très longues,on ne peut s'attendre à ce qu'un laboraratoire quelconque puisse trouver des résultats en deux mois.
A noter qu'un treillis de SiO2 recouvert de platine assure la recombinaison des gaz,ce qui évite les rajouts d'eau lourde.

-Electrolyse principale.
Elle se fait avec la cathode preparée comme indiquée ci-dessus et avec une anode en platine.L'intensité est maintenant de 500 à 600 mA/cm2.L'expérience dure de quelques dizaines d'heures à au moins dix jours.L'électrolyte est LiOD à 0,1 M.Les gaz ne sont pas récupérés.

-Résultats en tritium.(Article soumis à NATURE,Doc n° 1)
Il trouve une production de tritium dans huit cellules, correspondant au même lot de palladium.Ce tritium est recueilli dans l'électrolyte.Initialement,c'est le labo du Pr. Wolf qui a fait les mesures,mais Bockris vient de s'équiper d'un appareil commercial utilisant un liquide scintillateur type POPOP.Par souci de rigueur,des échantillons d'électrolyte et des électrodes ont été envoyés en analyse à Los Alamos,Batelle,Argonne,General Motors,

Livermore.(En ce qui concerne les analyses faites à Los Alamos,voir notre C.R.sur Los Alamos). L'augmentation de l'activité dans la cellule est mesurée au cours du temps et semble spectaculaire,passant de 200 à sept dix puissance cinq coups/minute en douze heures.Ce phénomène a été observé sur sept cellules sur neuf.Une expérience,où l'on a recueilli les gaz pour les recombiner sur un catalyseur au platine,a montré qu'il y avait dans l'eau formée une concentration de tritium 10 à 50 fois plus grande que dans l'électrolyte,ce qui prouverait que la majorité du tritium passe dans les gaz.Un examen de la surface de la cathode montre qu'il n'y a plus d'atomes de palladium en surface,(Voir visite labo Martin),mais que des dendrites,sans doute de nickel sont apparus.Une explication proposée est que la formation de dendrites permettant d'atteindre des champs électriques élevés associés à des différences de potentiel de 10 kV,conduit à des réactions nucléaires en surface.

-Résultats en énergie.(Art.accepté par Elec.Acta,Doc n° 2)

On trouve un surplus d'énergie de 3 à 30 % mesuré par rapport à (V-1,54)xI,cela sur trois cellules sur dix.Il importe d'avoir une évaluation assez précise de la recombinaison.Cette évaluation a été faite sur deux cellules et on a trouvé un chiffre compris entre 1 et 2 %. Cependant,les mesures ne sont pas faites sur les cellules ayant produit de l'énergie .Un montage est en cours pour cela.Il est à noter que les cellules sur lesquelles on a mesuré une production d'énergie ne sont pas les mêmes que celles sur lesquelles on a mesuré une production de tritium.L'énergie associée à la production de tritium dans l'électrolyte ne serait pas détectable.Si on tient compte,toutefois,du tritium perdu dans les gaz,il faudrait multiplier ces chiffres par 50 et la chaleur produite serait alors mesurable.

-Essais d'explication de l'énergie dégagée.

Dans un article détaillé,(Doc n° 3),le groupe analyse les principales possibilités de dégagement d'énergie autres que nucléaire.Il en conclue à l'impossibilité pratique d'expliquer les phénomènes autrement que par réactions nucléaires.

-Erreurs à éviter.

Dans un document à l'usage des visiteurs (Doc. n° 5), Bockris résume les erreurs à éviter,selon lui.Nous retiendront,en particulier:

- Un strict respect du protocole de chargement, avant électrolyse principale,
- L'utilisation de palladium très pur,type Johnson Mathey,
- La proscription de tout contact avec de l'eau ordinaire,
- Pour les mesures de tritium,l'assurance qu'il n'y a pas chimiluminescence.

-Travaux futurs.

Pour la suite,il est envisagé de travailler toujours en cellule fermée avec recombinaison par catalyseur,ainsi que d'avoir une gestion automatique des électrolyses afin de pouvoir en traiter un grand nombre à la fois.

VISITE DES LABORATOIRES DU PROFESSEUR MARSH.(Texas A & M Univ.)

Nous avons rencontré:

Pr. Marsh,
Dr.Gammon,

Le laboratoire de Marsh fait des expériences d'électrolyse soit seul,soit en collaboration avec le laboratoire du Dr. Martin.Notons que le laboratoire de Marsh est spécialisé dans les mesures thermodynamiques et qu'il travaille sous contrat pour le N.B.S.

Le groupe a essayé de mesurer des excès de chaleur.Sur 20 cellules,une seule a donné des excès de chaleur et l'on n'a pu,depuis,reproduire ce résultat.Notons que le palladium utilisé pour ces expériences était d'origine différente de celui utilisé par Bockris.Il a été,soit utilisé tel quel,soit recuit sous vide.Les tiges de palladium ont un diamètre variable de 1 à 2 mm et une longueur de 10 cm.Les cellules sont plongées dans un bain et les températures de la cellule et du bain sont maintenues constantes par des résistances.Les gaz ne sont pas recueillis et l'ensemble est fortement agité.On n'a jamais vu de tritium.(Mesure par Wolf).

Le problème de la recombinaison est examiné très attentivement et de nouvelles expériences,particulièrement soignées,sont en préparation pour préciser ce point.

VISITE DU LABORATOIRE DU DR.MARTIN.(Texas A & M Univ.)

Il a mesuré les états de surface des cathodes par sonde Auger.(Doc. n° 6).On examine d'abord la surface du palladium brut du fournisseur,puis ses états successifs.On voit l'influence du recuit qui fait diminuer fortement les pics de C,S,l'influence de l'electrolyte LiOD.Après électrolyse,le pic caractéristique du palladium disparait tandis qu'augmentent ceux du carbone et de l'oxygène.La nature de la couche superficielle est encore mal connue,mais le lithium y est peut être présent sous forme oxyde comme des expériences au SIMS le montrent.Une couche de plusieurs centaines d'angtröms de lithium dont l'épaisseur exacte est difficile à quantifier seait présente sous forme éventuelle d'oxyde.

A noter que son palladium provient d'un fournisseur différent de celui de Bockris.

ISITE DU LABORATOIRE DU PROFESSEUR WOLF.(Texas A & M Univ.)

En l'absence du Pr. Wolf,nous avons rencontré le Dr.Shoemaker qui nous a fait visiter le laboratoire de détection des neutrons.

Le Dr.Wolf a annoncé qu'il avait obtenu à la fois du tritium dans huit cellules et des neutrons dans trois.Une cellule produit à la fois du tritium et des neutrons mais pas dans le rapport auquel on peut s'attendre.Les cellules d'électrolyse qu'il utilise sont fournies par le laboratoire de Bockris.

Le système de détection des neutrons comporte un compteur constitué d'un PM et d'un scintillateur NE213,le tout blindé par un empilement contre les neutrons externes.Il y a un veto actif contre les cosmiques,mais loin d'être dans quatre pi et constitué de scintillateurs sur le toit.Ce veto serait actif sur 1 microseconde.Le signal le plus important a été de 2,7 coups/minute sur un bruit de fond de 0,9 coup/minute.Nous n'avons pas vu le spectre PSD correspondant.Le PSD est mesuré par un tiroir allemand.On ne connait pas la résolution en énergie ni le seuil.La "preuve" que le signal vu venait de la cellule a été obtenu en vérifiant qu'il chutait à presque 0 quand la cellule était éloignée à 12,5 cm du détecteur au lieu de 1,3 cm.Les calibrations ont été faites avec une source Pu-Be et Co60.

VISITE DU LABORATOIRE DU PROFESSEUR APPLEBY.(Texas A & M Univ.)

Nous avons rencontré,outre le Professeur Appleby,le Dr. Jalbert.

Ce laboratoire est spécialisé dans la microcalorimétrie. Le microcalorimètre utilisé est du type à deux cavités identiques,l'une étant vide et servant de référence,toutes deux immergées dans un container,avec mesure des différences de températures entre les deux cavités et entre les cavités et le container (Voir Doc.n° 7,déjà édité à Santa Fé).Les sensibilités sont de 3 à 5 microwatts entre les deux cavités et de 100 microwatts entre cavités et container.

Les cellules d'électrolyse sont en inox,de dimensions 5X5 cm.Il n'y a pas de courant de fuite,la fluctuation en tension ne dépasse pas 30 millivolts.Le palladium est un fil de 0,5 à 1 mm de diamètre sur 10 cm de long ou bien des sphères de 2 mm de diamètre.L'anode en platine est un fil de diamètre 0,5 mm et de 10 cm de long.La cellule contient de 7 à 8 millilitres d'électrolyte LiOD à 0,1 M .Les blancs sont faits avec LiOH dès le départ ou en remplacant par NaOD en cours d'électrolyse,(mais pas dès le départ). Un autre blanc est obtenu en remplaçant la cathode en palladium par une en platine.L'électrolyse démarre avec une cathode non préchargée,à une intensité de 60 mA/cm2 pendant 50 heures,puis on monte à 600 mA/cm2.On utilise couramment du lithium naturel.L'utilisation de lithium 6 ne change pratiquement pas l'état de régime,mais permet d'y accéder légèrement plus vite,sans doute parce que la vitesse de diffusion de Li6 est légèrement plus grande.Le sodium est de qualité nucléaire.Les gaz se dégagent librement.Le générateur est à courant constant.Les dégagements de chaleur sont reproductibles qualitativement,pas quantitativement sur toutes les expériences.Le dégagement d'énergie est de l'ordre de 3 à 30 % au maximum par rapport au produit (V-1,54)xI.Le recuit du fil permet d'obtenir plus rapidement le début du dégagement d'énergie.A noter,cependant,que les excès d'énergie restent faibles en valeur absolue et l'ordre de quelques milliwatts après la formation de l'hydrure.La possibilité de les attribuer à la recombinaison semble exclue par les mesures de bilan de gaz effectuées,qui conduisent à 1 à 2% maxima.

La recherche d'hélium dans la cathode,faite au spectro de masse,donne des limites supérieures de dix puissance neuf en atomes d'hélium 3 et dix puissance huit en hélium 4.

On a vérifié qu'aucun excès de chaleur ne peut provenir de l'oxydation de la cuve en inox.

Actuellement,le groupe étudie une cellule fermée avec recombinaison totale des gaz par catalyse sur un treillis de SiO2 recouvert de platine.

VISITE DU LABORATOIRE DE LOS ALAMOS.

Nous avons rencontré:

Reed Jensen,directeur,
Malcolm Fowler,
Anton Mayer,adjoint de Menlove,
Shermann,
Shimson Gottesfeld,de l'équipe Storms,
Edwards Garcia,
Paciotti,
J.Solem.

Il y aurait,au total,de 20 à 30 personnes impliquées plus ou moins dans la Fusion froide.

I/ Expériences en Gaz.(Menlove,Paciotti,Garcia,Fowler,Mayer)

Ces expériences sont décrites dans le document n° 8 ,préprint rédigé des transparents fournis à Santa Fé,9,10).Nous le complétons par la description orale qui nous a été faite.

Dans l'expérience exposée à Santa Fé,la bouteille contenait un mélange de divers matériaux,titane pur, alliage de titane,palladium,etc,sous formes diverses. Depuis,ils ont essayé d'isoler le matériau responsable des bursts.Il apparait que des résultats beaucoup plus reproductibles sont obtenus avec du titane industriel, type TA6V.(2% d'étain,6% de V,6% d'Al). Cet alliage comporte une proportion importante de phase béta.On a essayé divers autres métaux dont une cathode en palladium.

Le dispositif de mesure est constitué de 16 compteurs à Hélium 3 en couronne,entourés de modérateur,et reliés à un même ampli.Le shaper utilisé ne permet pas de mesurer la charge de l'impulsion.L'efficacité typique aux neutrons est de 30 %.Le bruit de fond typique de 0,24 Hertz sur les 16 compteurs.Après chaque impulsion,une porte de 128 microseconde est ouverte et donne la multiplicité d'un burst éventuel.Cette mulktiplicité est aussi mesurée avec une porte de 16 microsecondes.La comparaison des deux a permis de dire qu'il existait des bursts de moins de 5 à 10 microsecondes et de multiplicité de 10 à 80.Il n'y a pas de veto contre les cosmiques,mais ceux-ci sont identifiés comme donnant un signal surune couronne extérieure de six compteurs à He3 dont l'efficacité est 30 fois plus petite que la couronne des 16 compteurs pour des neutrons provenant ducentre. L'efficacité d'un tel dispositif n'est pas évidente mais semble justifié par le fait qu'on ne voit pas de bursts avec des blancs.

On met de 100 à 500 grammes d'alliage de titane sous forme de copeaux,pastilles,éponge,dans une bouteille en inox de 25 cm de haut,sur 5 cm de diamètre.Ce titane est chauffé à 200°pour dégazage sous vide à dix moins cinq torrs.Puis on introduit le deutérium à une pression de 40 à 80 atmosphères.La bouteille est alors refroidie à -190°C puis laissée se réchauffer.On peut faire jusqu'à dix cycles.En fin d'expérience,la pression de deutérium a beaucoup diminué.Notons que l'on cherche a avoir toujours un excès de métal par rapport au deutérium.

On a mesuré quelques émissions acoustiques.Il n'y a pas de mesure systématique de bruit de fond sur les bouteilles vides,mais sur deux bouteilles qui n'ont pas donné de neutrons,on n'a pas trouvé de bruit.Lorsque les bouteilles sont chargées en alliage de titane et deutérium,il n'y a aucune corrélation entre bruit et émission de neutrons.Sur certaines bouteilles transformées en chambre à ionisation,on a pu déceler une émission d'électrons.

Des bouffées de neutrons ont été obtenues pour 15 échantillons sur 30,à des instants divers.Trois bouteilles ont donné une émission continue de 3 à 4 fois sigma durant 10 heures alors que les bouteilles témoins ne donnent rien.A noter que sur trois essais en mélange (D+H), un seul a donné des neutrons.Il n'y a pas eu d'essais en hydrogène pur.

Les bouffées de neutrons sont de 30 à 40,quelquefois de 15 neutrons.Elles ne sont pas reproductibles,elles durent moins de 5 à 10 microsecondes.Le recyclage ne donne pas forcément des neutrons,mais,inversement,si on n'obtient pas de neutrons la première fois,on peut en obtenir la seconde.L'émission cesse toujours après quelques cycles.On a vu également des bursts avant tout refroidissement.Il n'y a pas de burst si l'hydruration du titane se fait préalablement à l'extérieur avant les mesures,ou si la pression de deutérium n'est que de deux atmosphères.

Actuellement,on démarre une expérience de chargement de titane par le mélange D+T.

II/ Expériences d'électrolyse.(Fowler)

En plus d'une expérience d'électrolyse du type Jones déjà décrite à Santa Fé et qui donne des bursts de neutrons,un deuxième type d'électrolyse a été étudié.Les électrodes sont élaborées sur place.Le palladium est refondu sous vide,mais pas à l'arc.On a réalisé ainsi des tiges de 4 mm,ensuite martelées.On n'a pas obtenu de résultats positifs jusqu'à présent.

Les chercheurs du groupe s'orientent d'une part vers l'amélioration des compteurs neutroniques,d'autre part vers des mesures calorimétriques.

III/ Expériences d'électrolyse.(Storms,décrite par Shermann)

Le groupe fabrique ses électrodes par fusion à l'arc.Ils ont essayé plusieurs alliages,palladium pur élaboré à partir d'une poudre de Johson-Mathey,mélanges palladium rhodium (0,9 Pd,et 0,1 Rh en atomes),puis laminage jusqu'à 3 mm.On cherche à fabriquer un produit comportant de nombreuses dislocations.Ils ont obtenu du tritium dans une cellule (Sur 16 au total)où il y avait des poisons,type thio-urée.(Pour une électrode contenant 0,1 de rhodium,et 0,038 de lithium).Le rapport D/Pd est obtenu par pesée et par mesure des dimensions.Il peut atteindre 1,2 en 250 heures.

Les mesures de tritium sont très élaborées:élimination des sels de lithium pour éviter la fluorescence,redistillation de l'eau.Ils ont pris le spectre complet d'émission.A noter que ce laboratoire,qui parait très compétent dans le domaine tritium,est également celui qui a mesuré le tritium produit dans les expériences de Bockris.A ce sujet,Shermann nous précise que les électrodes ayant produit du tritium chez Bockris ont été dégasées sous vide à 800 degrés avant électrolyse.Elles peuvent donc difficilement être soupçonnées d'avoir contenu du tritium avant l'électrolyse.Néammoins,leur analyses sont en cours.

Les électrolyses de Storms ont montré un enrichissement en tritium d'un facteur 20.

IV/ Expériences d'électrolyse.(Gottesfeld)

Ce groupe fait des électrolyse à la P. et F.Il déduit le chargement en deutérium par mesure de la résistance.

Les neutrons sont détectés à la fois par une couronne de compteurs à He3 et par un scintillateur liquide à NE213,placé au centre avec la cellule.(Voir le document N°11).Les gammas sont détectés par un cristal BGO et un germanium haute pureté.Le compteur à NE213 semble avoir vu un signal compatible avec des neutrons de 2,4 Mev mais les He3 n'ont rien vu!Réciproquement, les compteurs à hélium ont compté beaucoup pendant un certain temps tandis que le NE213 restait muet.Il y a donc artefact quelque part.

Les expériences calorimétriques,à la précision de 80 milliwatts sur 1,5 Watt,n'ont rien donné,pas plus que les mesures en tritium.

V/ Théorie.

-Calcul du rapport de branchement des réactions D-D
Le calcul a montré qu'aux très basses énergies relatives,les deux réctions D-D ne pouvaient différer de plus de 40 %,en faveur de la réaction productrice de neutrons.Ce résultat ne va pas dans le sens des résultats des expériences de fusion froide.

-Réaction D+T à basse énergie.
Les réactions D+D restent prépondérantes par rapport à D+T à basse énergie,le recoupement s'effectuant entre 200 et 400 Kev.

-Interprétation théorique des résultats des expériences de fusion froide.Los Alamos n'en a pas pour l'instant.

VISITE DU LABORATOIRE DU PROFESSEUR HUGGINS.(Stanford)

Nous avons rencontré le Professeur Huggins ainsi que le Dr Martha SCHREIBER,et Turgut GÜR.Par ailleurs, au déjeuner et l'après midi,trois chefs de projets de l'EPRI se sont joints à nous.(MM.J.CHAO,T.SCHNEIDER,R.WEAVER)

Le Professeur Huggins nous indique qu'il a obtenu une réussite systématique dans la production d'énergie par électrolyse.La dispersion dans la production d'énergie est de l'ordre de deux,sur cinq expériences.Les excès d'énergie sont mesurés en absolu par rapport au produit VxI,sans retrancher au potentiel la valeur 1,54 volt de l'électrolyse,ce qui exclue les erreurs possibles sur la recombinaison.L'excès maximum mesuré est de 14%,et,bien entendu,nettement plus si on enlève la valeur du potentiel de 1,54 Volt.

Le protocole expérimental est très précis:

- L'anode est un fil de platine enroulé au diamètre intérieur de la cellule pour que la surface apparente de l'anode soit supérieure d'un facteur 100 à celle de la cathode afin que la surtension anodique reste nettement inférieure à celle cathodique.
- La cathode en palladium est obtenue sous forme d'un nodule ayant la dimension d'un gros grain de café par dix fusions successives au four à arc sous argon,jusqu'à ce que le plasma soit de couleur stable.La sole est en cuivre refroidie par eau.Une première opération consiste à fondre du zirconium pour purifier l'argon,comme getter.On n'est toutefois pas sûr d'avoir éliminé le carbone du palladium,car,à la fabrication initiale,le fournisseur a utilisé un creuset en graphite.
- L'assemblage se fait en BAG en azote sec.L'eau lourde est fournie en ampoules scellées.La cathode est reliée au générateur par un fil d'or enroulé en croix tout autour.A noter que si la cathode est sortie à l'air,on n'obtient jamais de résultats positifs,l'hydrogène empoisonnant le palladium.
- La cellule est scellée à la cire.Elle est placée dans un bain d'huile et on mesure la différence de température entre la cellule et le bain.L'ensemble est fortement agité pour homogénéiser la température de la cellule.
- L'électrolyte est LiOD à 0,1 M.(20 grammes)

Les expériences sont faites sans préchargement en faisant varier les puissances injectées de 2 à 10 watts.Les résultats montrent qu'après deux jours en moyenne,il apparait une production d'énergie qui dure au moins 90 jours.Les excès d'énergie sont mesurés par les T.Concrètement,on effectue d'abord un étalonnage par une résistance dans le bain pour tracer la courbe T en fonction du produit VxI dans la résistance.On obtient une droite passant très près des points expérimentaux.Typiquement,une élévation de température de 10° correspond à 10 Watts. La température est mesurée à 0,01 ° près.Lorsqu'on passe à l'électrolyse en utilisant de l'hydrogène,on obtient une droite située sous la droite d'étalonnage,l'électrolyse absorbant de la puissance chimique.Cette droite correspond à une exothermicité inférieure à 7 % pendant toute la mesure.Si,maintenant,au lieu d'utiliser de l'hydrogène,on fonctionne au deutérium,la courbe de T en fonction du produit VxI démarre comme pour l'hydrogène,mais,au bout d'environ deux jours,elle passe au dessus de la courbe d'étalonnage.(Figure jointe).Ce qui s'interprète par une génération d'énergie.

Pour la suite,Huggins utilisera des cellules avec recombinaison des gaz.Il essaiera de mesurer le tritium formé.Enfin, il utilisera un système de gestion automatique de plusieurs cellules à la fois,avec enregistrement en continu des paramètres.

VISITE AU DOCTEUR GARWIN.(San Diego)

Le Dr.R.Garwin,professeur de physique à Colombia University et research fellow au Centre de recherche d'IBM,a été assez sceptique sur la réalité de la Fusion froide et ne s'en est pas caché.(Voir document n°12,"Nature",vol 338,20 April 1989).Par ailleurs,on sait qu'un panel vient d'être organisé par le D.O.E. à Washington sur la Fusion froide.Le Dr. Garwin a été membre de ce panel.A ce titre,il a été amené à examiner très officiellement les travaux récents des laboratoires concluant positivement à la réalité du phénomène.Nous allons examiner successivement ses diverses critiques.

I/ Dégagement d'énergie au labo Huggins.(Doc. n° 13)

Garwin remarque que la conclusion qu'il existe un excès d'énergie est entièrement basée sur la valeur du coefficient K d'étalonnage,K mesurant le rapport de la variation de température à la puissance fournie par la résistance.Or ce coefficient pourrait très bien varier avec la température.Ainsi,la courbe lissée de Huggins pourrait recouvrir des variations de l'ordre de 5 à 7 %.Le même problème peut se présenter lors de l'électrolyse.Il en résulte que les excès d'énergie,mesurés par les différences,seraient porteurs d'une erreur double,de 10 à 14 %.

Garwin évoque aussi la variation de résistance d'un facteur deux entre l'électrolyte en H et celui en D.

II/ Dégagement d'énergie au labo Appleby.

Ici,évidemment,Garwin lui reproche de déduire 1,54 volt pour calculer l'excès d'énergie.Garwin dit que la valeur de 1 à 2 % de la recombinaison demande à être mieux assurée.Compte tenu de la faible valeur absolue de l'excès d'énergie,l'imprécision en valeur absolue pourrait être importante.Garwin pense que,dans ces conditions,les excès mesurés pourraient n'être que du bruit de fond.

III/ Dégagement d'énergie chez Pons et Fleischmann.

Garwin estime que les calibrations pour la mesure de chaleur sont très mauvaises et qu'en outre,aucune mesure sérieuse de recombinaison n'a été faite.

IV/ Dégagement de tritium chez Bockris.

Garwin calcule aisément qu'avec le tritium trouvé chez Bockris,la deuxième réaction D-D produirait beaucoup de neutrons qui n'ont manifestement pas été vus.De plus,en supposant qu'il n'y ait que la seule réaction D-D productrice de tritium,il dit que le proton,au cours de son ralentissement,donnerait des gammas par diffusion inélastique et bremstrahlung sur le noyau de palladium.Selon un calcul de D.Bailey,ceci correspondrait à dix moins sept gammas par proton,(Doc. n° 14) entre 373 et 556 keV et l'on détecterait,pour dix puissance onze tritons par seconde,environ dix puissance quatre gammas par seconde.Ne voyant pas d'explications à la production de tritium,Garwin estime donc qu'il s'agit d'un artefact.

V/ Bursts de neutrons.

Il estime que la plupart des bursts trouvés pourraient être dus au bruit du compteur ou à d'autres évènements sans rapport avec l'expérience.Il nous donne un exemplaire (Doc n° 15) du rapport de la Sandia à ce sujet.Il pense qu'il faut une certaine redondance dans la détection des neutrons.Trois séries de compteurs indépendants,sans compter une bonne protection contre les cosmiques permettrait de conclure à l'existence de bursts avec une haute probabilité.

AUTRES TRAVAUX SIGNALES EN FUSION FROIDE.

I/ Travaux récents de Pons et Fleischmann.

D'après Appleby,P. et F. auraient obtenus des dégagements d'énergie continus de 100 % de VxI,avec des pointes à 1000 % après 60 jours,associées avec une ébullition brutale.

D'après Huggins,également des excès continus d'énergie de 15 %,puis des bursts de 50 à 100 fois plus élevés pendant deux jours représentant 11 MJ par mole de Pd, puis de nouveaux bursts durant quinze jours,où la température de la cellule passe de 32 à 54 °C.La montée en température a lieu neuf heures après une chute de tension de 4,2 à 3,6 volts.

II/ Travaux de Glen Schoessow,à Gainesville,Univ.de Floride.

On signale qu'il aurait obtenu par électrolyse,simultanément des neutrons à 20 sigmas,des gammas,de la chaleur au taux de 60 % de VxI.

III/ Travaux du Dr. Wadsworth,en Utah.

Il aurait obtenu des résultats identiques à ceux de P. et F.,avec des excès de chaleur de 100 fois VxI et des pics d'énergie intégrée de 0,2 à 1,6 Mégajoule. La durée des pics est variable autour de 90 minutes.

DISCUSSION GENERALE.

Production de neutrons.

La production d'un fond continu de l'ordre de 3 ou 4 sigmas n'est plus mentionnée pour les électrolyses.Los Alamos la mentionne pour quelques expériences en chargement gazeux de titane.Cela nous semble insuffisant pour affirmer l'existence du phénomène,étant donné la sélectivité du compteur.Quand aux bursts,les critiques de Garwin et de la Sandia nous semblent particulièrement valables.Les expérimentateurs,même à Los Alamos,ne sont pas à l'abri de déclenchements intempestifs.On peut néammoins penser que toutes les mesures neutroniques ne sont pas dues à des artefacts et que quelques unes correspondent à un phénomène intéressant.

Il est absolument nécessaire que les expérimentateurs utilisent des compteurs avec une certaine redondance,soit en ayant des séries de compteurs indépendants,soit en ayant des signatures multiples et une protection active contre les cosmiques.L'installation du C.E.A. au Bugey est exemplaire de ce point de vue.Notons que les efforts actuels de Los Alamos vont également dans ce sens.

Production de tritium.

Rappelons que le laboratoire de Bockris,au cours de huit expériences d'électrolyse,trouve des quantités de tritium de trois à cinq ordres de grandeur supérieures à celles introduites au total par l'eau lourde de l'électrolyte.Ces valeurs ne concernent que l'électrolyte.Dans l'eau obtenue par recombinaison des gaz dégagés,les concentrations en tritium seraient de 40 à 100 fois plus importantes que dans l'électrolyte,ce qui,compte tenu des volumes électrolysés,correspond à une production totale de tritium de 100 fois celle mesurée dans l'électrolyte.Ces mesures de tritium ont été confirmées sur plusieurs échantillons par cinq laboratoires,dont Los Alamos où des purifications poussées de l'eau et le relevé du spectre de tritium semble exclure toute possibilité d'erreur d'identification de la radioactivité mesurée.

Une conclusion inévitable de ces expériences est que le tritium provient de la cathode.En effet,l'électrolyse d'une eau tritiée donne,dans tous les cas,un gaz,donc une eau de recombinaison appauvrie en tritium par rapport à l'électrolyte de départ.Aucun phénomène physico-chimique connu ne saurait produire l'enrichissement observé du gaz par rapport au liquide ,sans compter les problèmes de bilan.De ce fait,l'électrolyte peut être tritié par échange avec le gaz qui se dégage,ou par d'autres mécanismes,mais il ne peut être la source du tritium contenu dans le gaz,même par contamination volontaire de l'électrolyte,(introduction malveillante d'eau tritiée) ou contamination par les parois de la cellule.Par contre,la cathode pourrait être imprégnée de tritium avant l'électrolyse.Deux expériences de Bockris où les électrodes ont été dégasées à 800° sous vide pendant 8 heures donnent toujours du tritium.De toutes façons,les mesures en cours à Los Alamos trancheront sur ce point.

Les critiques que l'on peut faire à ces expériences sont encore multiples,par exemple:

-Les expériences ayant fourni du tritium en quantité importante ont toutes été faites avec le même lot de palladium,

-Deux expériences de production de tritium faites avec le même palladium que les sept autres mais avec des cathodes de 3 et 6 mm au lieu de 1 n'ont rien donné en tritium,

-Enfin,on ne mesure pas de neutrons associés à ce tritium.Il faudrait donc supposer que seule,la réaction donnant T + P existe,ce qui suppose des explications pas faciles.

Le tritium étant relativement facile à détecter, il serait impensable,néammoins,que l'on n'arrive pas à en trouver l'origine.Peut être l'enquête en cours sera-t-elle longue mais elle sera payante.On saura alors s'il s'agit d'un artefact,comme le pense Garwin,ou bien s'il y a vraiment un phénomène nouveau.

Production d'énergie.

Ce problème de l'énergie nous semble le plus important puisque c'est lui qui conditionne les applications recherchées.Garwin ne démontre pas que les résultats expérimentaux obtenus sont faux,d'autant qu'aucun dégagement d'énergie n'a été noté sans eau lourde.Il faudra une véritable enquête pour dénouer tous les facteurs qui restent actuellement mélangés et imprécis,en particulier le taux de recombinaison.Tout ceci prendra du temps.

Si l'on examine plus particulièrement les critiques de Garwin sur les expériences de Huggins,(Doc. n°13) on constate que seule la précision de la mesure de la différence des puissances peut être mise en cause.D'après les chiffres de laboratoire,une erreur de 2x5 %=10 % serait à envisager dans le pire des cas sur les excès d'énergie.Or, comme rétorque Huggins,il reste encore de la marge sur les excès puisque on a volontairement négligé la recombinaison. Quant à la variation de résistance de l'électrolyte ou autres,elles s'éliminent dans la différence comme l'indique Huggins dans sa réponse.(Doc. n° 13).

Il est intéressant de comparer les quantités de tritium mesurées chez Bockris aux quantités de chaleur excédentaires observées aussi à A & M dans un autre laboratoire et sur d'autres cellules.Nous les avons rapportées par le calcul à la même longueur d'électrode,les dégagements de chaleur étant donnés en milliwatts par centimètres carré.On trouve alors que si l'on tient compte du tritium à la fois dans le gaz et l'électrolyte,l'énergie excédentaire mesurée est de l'ordre de un à quelques Mev par atome de tritium,ce qui est l'ordre de grandeur des réactions nucléaires connues.

Matériaux.

Le problème des matériaux parait très important pour obtenir des résultats positifs et il y a accord sur ce point.

Pour le palladium,P. et F. n'utilisent que des lots provenant de Johnson Mathey avec lesquels ils ont un accord.Bockris s'approvisionne chez Texas Coin Exchange et aussi chez Johnson Mathey.Il note un changement de comportement selon les lots d'un même fournisseur..Huggins a récupéré un creuset en palladium,l'a broyé,puis fondu à l'arc por le purifier et mettre en forme.Il semble que ce soit ainsi qu'il obtienne la meilleure reproductibilité.

Pour le titane,l'utilisation d'un alliage comportant une fraction importante en phase béta semble favorable.On rappelle qu'à température ambiante,il est très difficile de faire rentrer de l'hydrogène dans du titane pur.

Au cours de nos discussions,nous avons évoqué la possibilité d'une transformation martensitique brutale d'un métal entre les réseaux cubique centré et cubique à faces centrées.Ces transformations pourraient relacher brutalement l'énergie emmagasinée durant l'électrolyse et également produire des différences de potentiel instationnaires très importantes.Cette possibilité reste à étudier.

Dans la mesure où la Fusion froide existe,et où elle serait dûe à un phénomène de Physique du solide,on comprendrait assez facilement sa sensibilité à la pureté, voire au dopage du matériau.

Panel de Washington.

Les auteurs du rapport du panel (Voir document n° 16) sont prudents à juste titre et l'on ne comprendrait pas que l'on lance un grand programme sur des expériences inexpliquées et non reproductibles.Par contre,la recherche d'explication des expériences actuellement présentées comme positives est considérée comme nécessaire. Ils estiment que le financement de cette recherche doit se faire dans le cadre des enveloppes financières habituelles.

En dehors du D.O.E.,les soutients financiers viennent des Universités elles-mêmes,de l'E.P.R.I.,de Général Motors,de la Welch Foundation,etc et suffisent pour l'instant à assurer les travaux de petites équipes.On peut être sûr,par ailleurs,que les Américains feront suffisamment de réunions sur le sujet pour que les choses avancent rapidement.

CONCLUSION GENERALE.

De façon générale, au cours de cette mission, nous n'avons pu mettre en défaut les logiques expérimentales susceptibles d'être à l'origine d'un artefact important. Cela tient, sans doute, à la qualité des interlocuteurs que nous avions choisis, tous reconnus comme des scientifiques de grande valeur par la communauté internationale.

Il n'empèche qu'il reste encore beaucoup d'hypothèques à lever et de vérifications pénibles et délicates à effectuer. Actuellement, on peut dire que si aucune porte n'est fermée, par contre aucune démonstration rigoureuse du phénomène de Fusion froide n'a été réalisée.

Peut être, la bonne solution consiste-t-elle à suivre les conclusions du panel de Washington en poursuivant les études à un niveau relativement modeste, et ce, jusqu'à nouvel ordre. Un examen plus précis des besoins à ce sujet serait souhaitable ainsi qu'un minimum de coordination.

LISTE DES DOCUMENTS RAPPORTES.

1) Observation of tritium production during electrolysis of heavy water samples using palladium wires.
   Packham,Wolf,McLain,Bockris.
   Soumis à "Nature"

2) Sporadic observation of the Fleischmann Pons effect.
   Kainthla,Velev,Kaba,Lin,Packham,Szklarczyk,Wass,Bockris.
   Accepté par Electrochem.Acta.

3) Eight chemical explanations of the Fleischmann Pons effect.
   Kainthla,Szklarczyk,Kaba,Lin,Velev,Packham,Wass,Bockris.

4) Lettre de Bockris à Maddox,Directeur de "Nature".

5) To Cold fusion visitors. Bockris.

6) Observation of the surface composition of palladium cathodes after D2O electrolysis in LiOD solutions.
   Mebratu,Rodriguez,Bothwell,Cheng,Lawson,McBride,Martin,Soriaga.

7) Evidence for excess heat génération rates during electrolysis of D2O in LiOD using a palladium cathode.A microcalorimetric study.
   Appleby,Srinivasan,Kim,Murphy,Martin.

8) Measurement of neutrons emission from Ti and Pd in pressurized D2 gas and D2O electrolysis cells.
   Menlove,Fowler,Garcia,Mayer,Miller,Ryan,Jones.(Los Alamos)

9) Manuscrit de la liste des expériences au gaz à Los Alamos.
   Garcia.

10) Extrait du livre:"Titanium and its alloys".

11) Investigations into the phenomenon of cold fusion.
   Gottesfeld,Springer,Zawodzinski,Garzon.(Los Alamos)

12) Consensus on cold fusion still elusive.
   Garwin,Nature,Vol 338,20 April 1989.

13) Lettre de Garwin à Huggins et réponse de Huggins.

14) Lettre de Garwin à Wolf et Roth.

15) Search for cold fusion in high pressure D2 loaded Ti and Pd metal and hydride.
   Schirber,Butler,Ginley,Ewing.(Sandia Lab)

16) Interim report of the cold fusion panel to the energy research advisory board.

17) Bibliography of selected papers on colf fusion.
   Stack.(Los Alamos)

18) A & M pursues fusion mysteries.
   Dallas morning news.

19) Cold fusion.The heat mechanism.
   AbuTaha.

20) A study of ways to achieve a D/Pd ratio near unity.
   Storms,Talcott.(Los Alamos)

ANNEXE.RENSEIGNEMENTS COMPLEMENTAIRES.

Financement des travaux.

- Pons et Fleischmann.
  - .University of Utah:5 millions $.
  - .O.N.R.: 400000 $.
  - .Contrat General Motors.
- Laboratoires Texas A & M à College Station..
  - .University of Texas.
  - .Welch Foundation.
  - .E.P.R.I. à raison de 100000 à 200000 $ par labo.
- Laboratoire de Huggins à Stanford.
  - .University of Stanford.
  - .E.P.R.I.,quelques 100000 $.

Prochaine réunion sur la Fusion froide aux U.S.A.

Il est question d'une réunion au début Septembre, organisée par N.S.F. et -ou- par E.P.R.I. à Washington ou à Palo Alto.Appleby en serait co-chairman avec Chew.

Collaboration possible avec les labos U.S.A.

-Ce problème a été évoqué avec Appleby qui nous a indiqué qu'il était prêt à envisager une collaboration avec les Européens selon des modalités à discuter,qui comporterait un volet financier et un volet technique.

-Bien que n'ayant pas vu Bockriss lui-même,il semble qu'une collaboration soit possible avec son laboratoire qui comporte de nombreux étrangers.

-Avec Los Alamos,un collaboration existe déjà puisque ses chercheurs (Notamment le Dr.Garcia) envisagent d'utiliser les installations du Bugey ou du Fréjus.Par ailleurs,ils ont une collaboration avec le C.E.A.pour des calculs de réactions nucléaires entre éléments légers.